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Refractomet Division

UNIVERSAL-CYCLOPS STEEL CORPORATION

Technical Report

Bridgeville, Pennsylvania

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AMC TR 7-827 (I)

AMC INTERIM REPORT 7-827 (I)
December 1960

TUNGSTEN SHEET ROLLING PROGRAM

PHASE I TECHNICAL PROGRESS REPORT

UNIVERSAL-CYCLOPS STEEL CORPORATION
REFRACTOMET DIVISION
BRIDGEVILLE, PENNSYLVANIA

Contract AF33(600)-41917

29 September 1960 – 29 December 1960

The state-of-the-art of tungsten sheet rolling is summarized and the industry capabilities are analyzed. Selection of alloys, consolidation techniques, and conversion methods are evaluated.

BASIC INDUSTRY BRANCH
MANUFACTURING METHODS DIVISION

AMC Aeronautical Systems Center
United States Air Force
Wright-Patterson Air Force Base, Ohio

ABSTRACT - SUMMARY
First Interim Technical Progress Report

AMC INTERIM REPORT 7-827 (I)
December 1960

TUNGSTEN SHEET ROLLING PROGRAM

Refractomet Division
Universal-Cyclops Steel Corporation

A state-of-the-art survey of tungsten sheet rolling has been completed. Industry capabilities as related to raw material availability, consolidation practices, and conversion methods have been thoroughly analyzed.

Presently domestic tungsten sheet is available in sizes less than one-half the width and less than one-fifth the length of that desired under the present contract. Thus, many problems are encountered in attempting to select the desired alloy for scale-up.

This survey indicates that two consolidation techniques should be initially followed, i.e., arc melting and powder metallurgy. For the arc-cast work pure tungsten is recommended as the alloy, while for the powder metallurgy work K-100 powder is recommended. Since a development program sponsored by the Bureau of Weapons is presently underway for evaluating powder metallurgy production of tungsten sheet, it has been suggested by Wright Field personnel that the work under this program be limited to arc-cast tungsten.

Several conversion techniques will be employed for initial breakdown of the as-cast structure. The survey indicates that the arc-cast material should be extruded and then forged or rolled directly. However, preliminary data indicated that direct forging can be accomplished and should be investigated.

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FOREWORD

This Interim Technical Progress Report covers the work performed under Contract AF 33(600)-41917 from 29 September 1960 to 29 December 1960. It is published for technical information only and does not necessarily represent the recommendations, conclusions, or approval of the Air Force.

This contract with the Refractomet Division, Universal-Cyclops Steel Corporation, Bridgeville, Pennsylvania, was initiated under AMC Aeronautical System Center Project 7-827 "Tungsten Sheet Rolling Program". It was administered under the direction of Mr. Hugh L. Black, Project Engineer, Metallic Material Branch, Manufacturing and Materials Technology Division, AMC Aeronautical Systems Center, Wright Patterson Air Force Base, Dayton, Ohio.

Messrs. L. L. France and W. J. Schoenfeld of the Refractomet Division were the co-project engineers in charge at Universal-Cyclops Steel Corporation. Mr. Schoenfeld was responsible for the arc cast portion of the work, and Mr. France for the powder metallurgy portion. The survey itself was conducted by Battelle Memorial Institute with Messrs. D. J. Maykuth, V. D. Barth, and H. R. Ogden carrying out the survey.

Since the nature of this work is of interest to so many fields of endeavor, your comments are solicited as to the potential utilization of the material produced under this contract. In this manner, it is felt that a full realization of the resultant material produced will be accomplished.

* *

PUBLICATION REVIEW

Approved by C P Mueller

C. P. Mueller
Technical Manager
REFRACTOMET DIVISION

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State-of-the-Art Report by
Battelle Memorial Institute

I. INTRODUCTION

Air vehicles moving through space at higher speeds will require air frames capable of sustained operation at elevated temperatures far in excess of those encountered by today's vehicles. These air frames will require surfaces which are capable of withstanding temperatures in excess of 2500°F. The refractory metals with their appropriate coatings possess the necessary properties to meet these rigid requirements. Due to the fact that temperatures above 3000°F are anticipated in the very near future, structural sheet materials of tungsten will be required.

The purpose of this contract is to establish the state-of-the-art of rolling tungsten and tungsten alloy sheet in order to advance the industry capability to economically produce sheet to the required quality and sizes. The overall objective is to develop new and/or improved techniques for rolling large size tungsten sheet. The measure of accomplishment will be the production of acceptable tungsten sheet 36" x 96" in thicknesses of .020", .040" and .063" in a flat condition with uniform properties.

In order to accomplish the aforementioned aims, the program is broken down into five phases which are summarized below.

Phase I – State-of-the-Art Analysis

The objective of this phase is to evaluate the current state-of-the-art of tungsten sheet rolling throughout the sheet mill rolling industry. Also the planning of a detailed program to satisfactorily accomplish the development effort required to advance this state-of-the-art.

Phase II – Ingot Process Development

The refinement of tungsten ingot production processes including the establishment of tests and testing procedures to insure satisfactory uniformity of tungsten ingots.

Phase III – Development of Rolling Operations

This phase encompasses a study of the breakdown of tungsten ingots to establish process parameters including an analysis of the processing variables. Further, the establishment of processing controls and test procedures for the controlled rolling of tungsten sheet will be accomplished.

Phase IV – Process Uniformity Verification and Post-Rolling Development

This phase will encompass the controlled rolling of tungsten sheet using the process developed in Phase III. In addition, post-rolling development will be accomplished in an effort to establish specification for the production run.

Phase V – Final Pilot Production

The production of 20 sheets of 36" x 96" sheet in thicknesses of .020", .040" and .063" will be accomplished. This production should prove sufficient to demonstrate the reliability of the developed process and verification of the uniformity of flat sheets produced using the developed techniques.

II. PHASE I ANALYSIS

The results of the state-of-the-art survey performed by Battelle Memorial Institute are as follows:

1. Both arc melted and powder metallurgical alloy candidates should be evaluated for ingot process development in Phase II.

2. Unalloyed tungsten should be selected as a candidate for the arc melted material.

3. K100 grade of doped tungsten powder should be selected as a powder metallurgy material.

The bases for these recommendations are fully established in the attached report by Battelle. Since a complete analysis is given in the Battelle section, no further justifications for these recommendations will be given here.

The initial work on this program will be directed toward producing unalloyed arc cast tungsten sheet rather than powder metallurgy sheet. This decision arises from the following:

1. The powder metallurgy method of producing tungsten sheet has received much more attention than arc cast material. Consequently, the processing methods for producing powder metallurgy tungsten sheet are much more refined.

2. It is felt by the Air Force* that the Bureau of Naval Weapons tungsten sheet rolling program is thoroughly investigating the powder metallurgy approach to the consolidation and fabrication of sheet from unalloyed tungsten and variously doped tungsten powders. The Air Force, therefore, thought it needless to duplicate that effort, as the results of that work program will be available for comparison with the results of work performed to produce sheet from arc cast material.

III. PHASE II WORK – INGOT PROCESS DEVELOPMENT

In presenting a program to completely evaluate the ingot process development, it is obvious that many variables must be isolated during this phase of the program. This necessitates the incorporation of various techniques and thus makes development of the process complete and variable.

The survey has shown that there are insufficient data established on alloys of arc cast tungsten to warrant a major investigation toward the production of sheet. It is anticipated that other programs will evolve arc cast tungsten alloys with substantial improvement over unalloyed tungsten, but it is not the purpose of this program to develop alloys. Unalloyed arc cast tungsten will, therefore, be utilized.

To obtain the program objectives of lowest practicable temperature of transition from ductile to brittle behavior, maximum consistency of grain structure and recrystallization behavior, freedom from lamination and other defects, and

*Private communication from WWRCEP to LMBML.

chemical homogeneity, arc melted tungsten has a much higher probability of success than powder compacted material. Unalloyed tungsten should be selected as the candidate for arc melting.

The actual melting of small developmental tungsten ingots presents no severe problems. Five-inch-diameter ingots up to twelve inches long can be made consistently and a standard process has been developed for this operation. Material losses in conditioning small ingots, however, are severe and a 50 to 60 per cent yield is considered normal. The major loss on these ingots is removal of the side wall rather than top and bottom cropping. By scaling up to larger ingots, the amount of side wall removed on a relative cross-sectional area basis will be much less. On production heats of molybdenum and molybdenum alloys, the normal yield ranges from 85 to 90 per cent.

Since the final sheets in this program will weigh approximately one-hundred and forty pounds, and assuming an overall yield of 20 to 30 per cent, ingots weighing approximately 500 pounds will be required. The primary effort in this phase will, therefore, be to improve yields on the ingot sizes presently available and to develop methods for producing large ingots. Methods of initial breakdown to a shape suitable for subsequent fabrication to sheet will also be investigated.

The important variables which will be investigated or held constant in this phase are as follows:

a. Powder Chemistry

b. Electrode compaction methods, configuration and density

c. Methods of joining electrodes

d. Ingot melting

e. Ingot evaluation

f. Initial breakdown

g. Evaluation of Initial Breakdown Methods

a. Powder Chemistry

Previous experience on all the refractory metals, including tungsten, indicates that the purity of the starting material affects both the melting conditions and subsequent fabrication procedures. In an effort to eliminate this variable within the program, one powder lot will be used for all phases. The powder lot selected will conform to the Universal-Cyclops' Powder Specification for Tungsten, WEP 61-3.

b. Electrodes

Powder conforming to the above referenced specification will be compacted into electrodes by isostatic pressing. This method of compaction has been shown to be the most satisfactory for producing uniform density bars. After pressing, all electrode bars will be sintered in hydrogen to the specified density. Dimensional tolerances and density will conform to the Universal-Cyclops' Tungsten Electrode Specification, WEB 61-3.

An extensive program on the effect of density on melting phenomena has been conducted using densities from 75 to 95 per cent of theoretical. No appreciable effect could be observed during melting and subsequent ingot evaluation. Lower density bars, however, are more difficult to join and also more subject to breakage. High density bars provide more material per unit volume in the electrode which is advantageous in electrode assembly. From the above information, it has been concluded that a 90 per cent density, as specified in the above referenced specification, will be utilized throughout this program.

Although several electrode configurations have been successfully melted, a round bar is easier to produce, handle and join, and all electrodes in this program will, therefore, be round.

c. Electrode Joining

Sintered bars up to two inches in diameter have been successfully butt welded in an inert atmosphere. Larger bars have been tapped and threaded; however, many problems were involved in this process. An investigation of the feasibility of welding large diameter bars (up to 4" diameter) will be conducted. A study of machining techniques will also be investigated. The criteria for evaluating a joint will be sufficient strength to hold the entire electrode weight during assembly and subsequent melting and the absence of a bow in the electrode across the joined area.

d. Ingot Melting

As previously stated, melting tungsten presents very little investigation, but the yields obtained are subject to great improvement. It has been observed in refractory metal melting that the electrode to mold ratio is very critical with respect to the side wall condition achieved, and it is considered that this is the first and most important consideration in improving the yield. Electrodes measuring 1.5, 1.75, 2.00, 2.25 and 2.50 inches in diameter will be melted into a 3.875 inch diameter mold. The ingots will then be machined to the maximum diameter which will provide a defect free surface. By this method, the optimum electrode to mold ratio can be determined. It is anticipated that five nominal sixty pound ingots will be required for this determination. Using the optimum electrode to mold ratio, five additional sixty pound ingots will be melted and conditioned.

Using the information developed on joining larger diameter electrodes, a four inch diameter electrode will be assembled and melted into a nominal nine inch diameter mold providing an ingot weighing approximately 900 pounds.

e. Ingot Evaluation

All of the above ingots will be evaluated by the following methods:

1. Chemical analysis to determine metallic and interstitial element levels.

2. Contact and immersion ultrasonic examination for the presence of voids and/or other defects.

3. Dye penetrant inspection of all surfaces to determine the presence of cracks.

4. Identification of process variables associated with arc melting and conditioning.

5. Establishment of quality control limits for processing through the ingot stage.

In addition to the above evaluations, destructive testing is required as a correlation of the results obtained by non-destructive testing and to establish specific variables which cannot be determined by other methods. Two ingots will, therefore, be further evaluated as follows:

One as cast ingot will be subjected to a high temperature homogenization heat treatment. This ingot and the second as cast ingot will be sectioned for the following determinations:

1. Metallographic examination including microscopic, macroscopic and fractographic studies to determine the presence of voids, concentrations of metallic elements and interstitials, grain size and other areas uniquely evaluated by this means.

2. Hardness surveys throughout the billet cross-section.

3. Chemical analysis to determine the distribution of metallic and interstitial elements.

The effect of the homogenization heat treatment will be made by comparing the data received from the above evaluations.

f. Initial Breakdown

Considerable experience has been gained on initial breakdown of tungsten ingots by extrusion. Most of this work, however, has been accomplished at temperatures and extrusion speeds above the range of large commercial extrusion presses. A scale-up to large ingots will require investigation to determine the best method of initial breakdown. The following study on small ingots is designed to provide information which will be useful in scaling up. Of the eight small ingots remaining after ingot evaluation, three will be extruded according to the schedule in Figure I. The remaining five will be direct forged as shown in Figure II.

g. Evaluation of Initial Breakdown Methods

The yield achieved is of primary interest in evaluating the initial breakdown method. Also of great importance is the method feasibility; that is, can this method be used with reliability in producing a consistent product.

As previously stated, extrusion has been used as the conventional approach to initial breakdown of tungsten ingots. Direct forging has, however, been successful on several tungsten ingots, and this method shows good promise of success in larger sizes.

All wrought material produced will be evaluated as follows:

1. The individual and group yields will be determined on the sheet bars produced.

2. Contact and immersion ultrasonic examination for the presence of voids, cracks and/or other defects.

3. Dye penetrant inspection of all surfaces to determine the presence of surface cracks.

4. Metallographic examination to determine grain size, degree of recrystallization, grain flow characteristics and other areas identified by this investigation method.

5. Hardness surveys to determine the degree of work and uniformity.

6. Correlation and identification of the process variables associated with initial breakdown techniques.

7. Establishment of process procedures and quality control limits in producing sheet bar.

Using the process procedures established, the one large ingot will be forged or extruded into a primary wrought product and evaluated under the quality control limits previously established.

The results obtained from the work of Phase II will provide optimum procedures for producing arc cast ingots and present a preliminary approach to initial breakdown techniques. After approval of Phase II, work on further refinement of initial breakdown techniques and primary rolling of sheet will commence. The objective in Phase III is to develop processing procedures and produce tungsten sheet 24" x 24" in gauges of 0.020", 0.040" and 0.063".

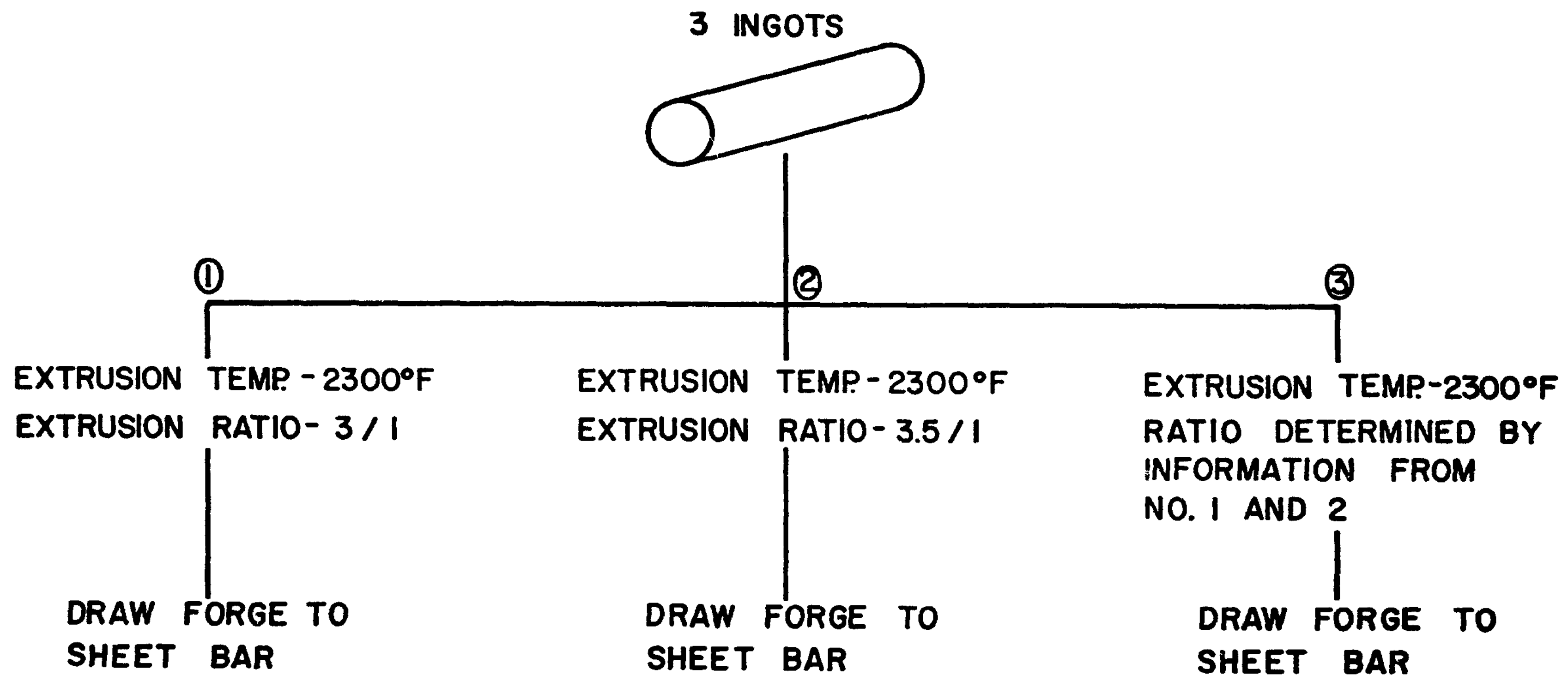
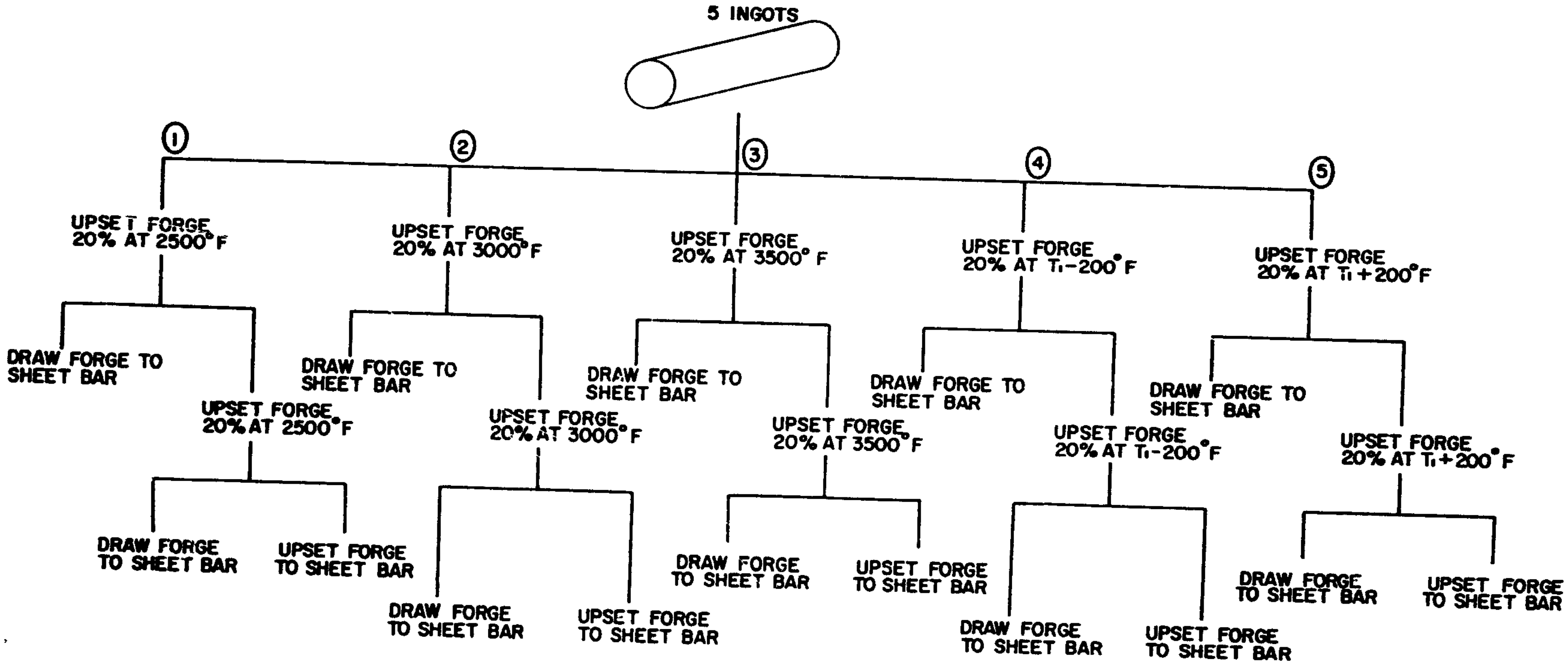


FIGURE 1

EXTRUSION EVALUATION OF ARC CAST INGOTS

RTA-0017



T_1–OPTIMUM TEMPERATURE DETERMINED FROM FORGING INGOTS 1, 2, & 3

FIGURE 2 FORGING EVALUATION OF ARC-CAST TUNGSTEN INGOTS

RTB-0002

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DEVELOPMENT OF NEW OR IMPROVED TECHNIQUES FOR THE PRODUCTION OF TUNGSTEN SHEET

A STATE-OF-THE-ART SURVEY

by

D. J. Maykuth, V. D. Barth, and H. R. Ogden

Battelle Memorial Institute

December 29, 1960

for

Universal-Cyclops Steel Corporation

for submission to

Manufacturing Methods Division
AMC Aeronautical Systems Center
Wright-Patterson Air Force Base, Ohio

on

Air Force Contract No. AF 33(600)-41917

FOREWORD

This report covers the work performed under Contract AF 33(600)-41917 from September 29, 1960, to December 29, 1960. It is published for technical information only and does not necessarily represent the recommendations or conclusions of the Air Force.

The state-of-the-art survey was conducted by Battelle Memorial Institute as a subcontractor to the Universal-Cyclops Steel Corporation on the above contract. The primary responsibility for planning and conducting this survey was centered in the Nonferrous Metallurgy Division under the direction of H. R. Ogden, Division Chief, with D. J. Maykuth, Assistant Division Chief, and V. D. Barth, Principal Metallurgical Engineer in the Powder Metallurgy Division assisting in this effort. The over-all guidance and valuable comments of Dr. R. I. Jaffee, Technical Manager of the Metallurgy Department, are also gratefully acknowledged.

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DEVELOPMENT OF NEW OR IMPROVED TECHNIQUES
FOR THE PRODUCTION OF TUNGSTEN SHEET

by

D. J. Maykuth, V. D. Barth, and H. R. Ogden

INTRODUCTION

By virtue of its high melting point and relative abundance, tungsten occupies a unique position among refractory metals for application in airframes, re-entry vehicles, and nozzles and vanes for rocket motors. Much progress has been made in developing massive tungsten parts that suit some of these applications. However, production applications for tungsten in thin sections have been hampered by the limited availability of sheet in the sizes and quality desired for advanced military aircraft.

In recognition of this problem, the Air Materiel Command recently awarded the Universal-Cyclops Steel Corporation U. S. Air Force Contract No. AF 33(600)-41917 for the "Development of New or Improved Techniques for the Production of Tungsten Sheet". This program involves five phases to run in chronological sequence as follows:

(1) Completion of a state-of-the-art survey on rolling tungsten sheet

(2) Ingot process development

(3) Development of rolling operation

(4) Process uniformity verification and postrolling operation development

(5) Final pilot production of 36 x 96-inch sheet in thicknesses of 0.020, 0.040, and 0.063 inch.

This first interim report represents the bulk of the Phase 1 effort. This survey was conducted by the Battelle Memorial Institute with the assistance and cooperation of the Universal-Cyclops Steel Corporation. The objectives of the survey were to assess the current state of the art in the rolling of tungsten sheet and to recommend the composition(s) of a tungsten sheet material or materials for evaluation in the Phase 2 effort.

In conducting the survey, use was made of a questionnaire and personal interviews as well as an extensive search of the literature and the Defense Metals Information Center.

The questionnaire used is reproduced in Appendix A to this report. This was mailed to approximately 140 organizations known or believed to have had experience in the production of tungsten or tungsten alloys or in converting compacts or ingots of these materials to sheet or other wrought forms. The complete list of organizations contacted is given in Appendix B in which the specific organizations visited are also designated.

Wherever possible, the data cited have been referenced to the pertinent Government reports or publications in which these have appeared. The opinions and recommendations given are the authors' interpretation of the total information gathered during the survey.

CONCLUSIONS AND RECOMMENDATIONS

The following conclusions were reached as a result of this survey:

(1) The only tungsten sheet materials which have a demonstrated production capability are unalloyed tungsten, the 1 and 2 per cent thoria alloys, and two proprietary doped grades, i.e., Types 218 and K-100.

(2) Each of these materials is being made by powder-metallurgical techniques which are essential, in all but the unalloyed grade, to obtain optimum properties through control of composition and dispersed particle size (i.e., thoria).

(3) The largest size of unalloyed tungsten sheet made commercially is 0.040 x 17 x 17 inches (one producer) and this producer is currently engaged on Bureau of Naval Weapons Contract No. NOw-60-0621c which has, as the ultimate objective, the production of 0.060 x 18 x 48-inch tungsten sheet using powder-metallurgical techniques.

(4) Thoriated tungsten sheet appears to offer the best prospects for obtaining a significant improvement in the strength of unalloyed tungsten at temperatures above about 3500 F*, without substantially decreasing the recrystallization temperature of tungsten or substantially lowering its melting point. However, the preparation of large-sized, thoriated tungsten sheet bars entails appreciably more difficulties than for unalloyed tungsten and the state of the art for rolling wide thoriated tungsten sheets is considerably less advanced. [See (3) above.]

(5) Of the two doped grades of tungsten being converted to sheet material, the greatest size capability has been demonstrated for the K-100 grade.

(6) The feasibility of producing tungsten sheet from consumable-electrode arc-melted unalloyed ingot has been demonstrated by the Universal-Cyclops Steel Corporation. Through the use of extrusion to break down the cast structure and forging to sheet bar, pilot sheet samples have been obtained which compare favorably in size to the largest of those now being made using powder-metallurgy techniques.

(7) No arc-melted tungsten alloys, in ingot sizes above 2 inches in diameter, have been converted to sheet material.

*This conclusion is based on the assumption that strength properties obtained in thoriated tungsten bar stock can be obtained in thoriated tungsten sheet.

(8) Generally, arc-melted tungsten product is characterized by a higher total purity than can be presently obtained by powder-metallurgy consolidation practices. The higher purity associated with the arc-melted product may be expected to contribute to greater ductility in this material at elevated temperatures. This has already been reflected in the successful use of lower rolling temperatures for arc-melted product (after extrusion and forging). Conversely, the higher purity may lead to a reduction in the recrystallization temperature.

(9) It is recognized that, in the ultimate scaleup of a rolling practice for arc-melted billet, some difficulty may be encountered by the lack of adequate heating facilities for extruding the large billets required.

On the basis of these conclusions, the following recommendations are made:

(1) It is felt that the presently active Air Force sheet rolling program should be confined to unalloyed tungsten sheet product, fabricated from arc-melted material. One reason for this stand is that the Bureau of Naval Weapons tungsten sheet rolling program, being conducted by Fansteel Metallurgical Corporation, is thoroughly investigating the powder-metallurgy approach to the consolidation and fabrication of sheet from unalloyed tungsten and variously doped tungsten powders. It therefore is thought needless to duplicate that effort, as the results of that work program will be available for comparison with the results of work performed to produce sheet from arc-cast material.

(2) It is recognized that somewhat higher strength at temperature, than available in tungsten, may be required for certain structural applications and some types of rocket nozzles. According to present knowledge, thoriated tungsten, a powder product, is the best example of such an alloy. However, the preparation of thoriated tungsten sheet bars entails appreciably more difficulties than for unalloyed tungsten. At such time as the Bureau of Naval Weapons program develops optimum techniques for producing powder-compacted sheet bars that can be fabricated to high-quality sheet, it may be a logical follow-up for an Air Force program to provide wide, powder-metallurgy, thoriated tungsten sheet.

(3) It is thought that by confining the AMC sheet rolling program to arc-cast unalloyed tungsten material, the state of the art will be more satisfactorily and rapidly advanced than by having the effort divided between a powder-metallurgy approach and an arc-casting process. To provide the sheet size requirements of the AMC program will require ingot sizes considerably larger than those heretofore converted to sheet material. This will introduce new problems in melting and primary breakdown. Also, there will be need to investigate the effect of numerous processing variables on the purity, mechanical, and physical quality of the sheet product. The objective of the program is not only to produce a given size sheet of minimum dimensional variation, and free of laminations and other defects, but to develop processes which will provide chemical and structural homogeneity,

the lowest possible ductile-to-brittle transition temperature and consistency of recrystallization behavior. If the aforementioned industry capability is accomplished by the AMC project, for unalloyed arc-cast tungsten, it will be possible at some future date to readily accomplish sheet production of arc-melted tungsten alloys indicated suitable from laboratory alloy-development programs.

(4) It is strongly recommended that unalloyed arc-cast tungsten be the material from which sheet is to be fabricated by the subject project.

SUMMARY

All tungsten sheet now being made commercially is prepared by powder-metallurgy techniques. In this process, tungsten powder with an average particle size in the range of 1 to 10 microns, is mechanically or isostatically pressed to sheet bar. Resulting bar densities of about 65 to 75 per cent of theoretical are obtained. The bars must then be densified to a minimum of about 85 per cent of theoretical by high-temperature sintering in hydrogen or vacuum. Sintering also has the desirable effect of purifying the powder, especially with regard to oxygen. The sintered product is then rolled directly to sheet, with or without the benefit of high-temperature forging to further improve densification.

Most of the producers' effort with tungsten as a sheet material has been given to rolling the unalloyed metal. The maximum size sheet now available from U. S. producers is 17 inches wide by 17 inches long at 0.040-inch thickness (one producer). This same producer is currently engaged in a pilot development program, for the Bureau of Naval Weapons, which has the ultimate objective of producing 3500 pounds of 0.060 x 18 x 48-inch tungsten sheet by powder-metallurgical techniques.

Aside from unalloyed tungsten, only four tungsten sheet "alloys" have been produced on a commercial or semicommercial basis. These include the 1 and 2 per cent thoria alloys, available from several producers, and two doped grades, Types 218 and K-100, available from two individual producers. Each of these alloys is being made by powder-metallurgical techniques. Production of the thoriated grades is appreciably more difficult than for unalloyed tungsten. At the present time, these as well as the Type 218 grade are not available as sheet in widths above about 4 inches. The K-100 grade has been rolled to sizes as large as 0.060 x 7 x 27 inches and 0.065 x 10 x 36 inches.

At the present time, equipment limitations, rather than technological limitations, appear to be the major deterrent to increasing the size (width) capability of powder-metallurgy sheet. The need appears especially critical for protective-atmosphere furnaces capable of heating large-size sheet bars and slabs to the temperatures required for (1) adequate densification, and (2) preheating for breakdown rolling.

Satisfactory techniques have been developed for the extrusion and forging of sintered tungsten and tungsten-alloy billets. For satisfactory recoveries of material in these operations, sintered billet densities at least as high as those for direct rolling

sheet bar are required. Neither extrusion nor forging is being used to convert massive, round sintered sections to sheet bar.

Experience in the consumable-electrode arc melting of tungsten is being accumulated rapidly. At least seven organizations have used this procedure to produce good quality unalloyed tungsten ingots in diameters of 4 inches or greater. Unalloyed tungsten ingots as large as 9 inches in diameter have been made. So far as tungsten alloys are concerned, production melting capabilities have been demonstrated for only a few binary tungsten-molybdenum compositions, most notably the 85W-15Mo alloy which is now being melted by several producers in ingot diameters up to 12 inches. Electron-beam melting of tungsten shows equally good promise for preparing tungsten ingots although present equipment is limited to a maximum tungsten-ingot diameter of 4 inches.

Aside from two notable exceptions, no successes have been achieved in direct forging or rolling large-diameter (greater than 2 inches) arc-cast tungsten or tungsten-alloy ingots. In both of these instances, the key to success appears to lie with the use of grain-refining additions, and reproducibility of these successes has not yet been demonstrated. Thus, at the present time, the most practical means of breaking down as-cast tungsten-alloy ingots is by extrusion.

Various facilities and organizations have successfully extruded arc-melted ingots of unalloyed tungsten and a variety of tungsten-base alloys. Most of this work has been done on an experimental basis. Nevertheless, sufficient progress has been made with unalloyed tungsten and a few tungsten-molybdenum alloys so that yields of better than 50 per cent can be consistently expected in extruding these materials to simple rounds.

Through the use of extrusion, the feasibility of producing tungsten sheet from arc-cast material has been demonstrated. In this development work, sheet in sizes up to 0.040 x 6-1/2 x 17 inches was obtained from portions of extruded bar after subsequent forging to sheet bar and rolling at 2300 F. This experience indicates that breakdown rolling of arc-melted sheet bar (after breakdown by extrusion and forging) can be carried out at appreciably lower temperatures than those required for the sintered product (2700 to 2900 F).

The ductile-to-brittle transition temperature of tungsten has been shown to be sensitive to such variables as grain shape and size, strain rate, and metal purity. While the tensile transition temperature of tungsten sheet has not been determined, the ductile-to-brittle bend transition appears to occur over the same temperature interval found for that of tungsten bars and rods in tension, i.e., about 300 to 850 F. In all instances, the lowest transition temperatures observed are for wrought or cold-worked material.

A fair amount of information has been generated concerning the effects of impurities on the properties of tungsten. Unfortunately, much of this remains qualitative in nature due to the general inability of present analytical techniques to accurately measure impurity elements in tungsten at levels below about 10 ppm. Nevertheless, it has been shown that variations in the interstitial content of tungsten, in the range of 1 to 20 ppm, are apparently not a factor in determining the degree of low-temperature ductility in tungsten, i.e., at temperatures around the ductile-to-brittle transition. Rather, variations in trace metallic impurities are suspected. Similarly, interstitial impurities, in the ranges normal for sintered product, appear to have little effect on the recrystallization temperature of tungsten while variations in trace metallics can apparently affect the recrystallization temperature by as much as 700 to 900 F. With the improvement of analytical techniques, it is conceivable that effects of very low concentrations of interstitial impurities will be more evident.

The tensile strength of tungsten at temperatures through 2500 F appears quite sensitive to processing variables. The effect of these becomes less marked with increasing test temperature and, above about 3500 F, the tensile strength of tungsten appears essentially independent of both the consolidation practice used and prior thermal history. However, at temperatures above about 2500 F, the type of consolidation practice appears to have a marked effect on the degree of tensile ductility obtained. Thus, reduction-in-area values for sintered product decrease drastically above this temperature while high values are retained in arc-melted product at temperatures at least through 4200 F. Several organizations have shown that arc-melted ingot normally contains appreciably less impurities than sintered and fabricated product made from the same material. These purity differences appear to offer the main basis for explaining the high-temperature ductility differences between these types of material.

Several dilute tungsten-base alloys, prepared and tested as bar stock, show significant strength advantages over unalloyed tungsten at temperatures to about 3500 F. At higher temperatures, the only addition shown to improve the strength of tungsten is thoria, in amounts of 1 to 2 per cent.

STATE-OF-THE-ART SURVEY

RAW MATERIALS

At the present time, all wrought forms of tungsten originate from a powder product. Hence, for purposes of the present report tungsten powder is considered as the principal raw material for tungsten sheet. Further, the starting material is considered to be pure unalloyed tungsten except for certain alloys where the as-reduced powder contains doping additives, a co-reduced metal such as molybdenum, or some other modifying agent added at the prereduction stage.

Unalloyed Tungsten Powder

The production of unalloyed tungsten powder follows fairly well standardized lines although many of the specific processing details used by the various producers are proprietary.

The major producers and/or suppliers of tungsten powder in the United States at the present time are as follows:

Belmont Smelting and Refining Works, Inc.
Cleveland Tungsten, Inc.
Fansteel Metallurgical Corporation
Firth Sterling, Inc.
General Electric Company
Metal and Thermit Corporation
Metals and Residues, Inc.
North American Phillips, Inc.
Reduction and Refining Company
Shieldalloy Corporation
Sylvania Electric Products, Inc.
Union Carbide Corporation
Wah Chang Corporation
Westinghouse Electric Corporation

The initial stages of extractive metallurgy have been discussed by several authors(1,2,3)* and are not of direct interest here. Accordingly, only those reduction procedures leading to the production of mechanically workable tungsten will be mentioned.

Commonly, tungsten-metal powder is produced by the hydrogen reduction of purified yellow tungstic oxide (WO_3). Ammonium paratungstate [$5(NH_4)_2O \cdot 12WO_3 \cdot xH_2O$] is an intermediate in the preparation of tungstic oxide, but may also be reduced with hydrogen to yield tungsten metal directly.

*Numbers in parentheses refer to reference list at end of report. Where references are not specifically noted, information has come from interviews and/or questionnaires, or other sources.

Tungsten compounds may be reduced to tungsten metal by other procedures, e.g., by carbon reduction, but these procedures are either not presently economical or the product is inferior with respect to particle size or purity for making wrought tungsten shapes.

Particle Size

The particle size of as-reduced tungsten powder is dependent on a number of variables. These include the physical size and purity of the original oxide, the reduction time and temperatures used, and the concentration of water vapor in the hydrogen reductant. Further, the reduction can be effected in either a single- or double-stage process, the latter being especially desirable in the production of finer particle sizes (i.e., around 1 micron in average diameter).

Details of oxide preparation and reduction schedules are largely proprietary in nature with each producer.

Although tungsten-metal powder is produced commercially in a wide size range, the preferred size range for consolidation to fabricable shapes extends from about 0.5 micron in diameter to about 10 or 15 microns. A size-distribution characterized as "fine" commonly ranges from 0.5 to 3 microns with the distribution weighed heavily around the 1-micron-diameter size. A medium-range powder commonly includes diameters from about 0.5 to about 5 microns with a larger percentage in the 2- and 3-micron particle sizes. A coarse powder may range from 0.5 to about 10 microns with heavier distributions in the 4- and 5-micron size. Figure 1 illustrates the particle-size distribution in four successively coarser mixtures offered by one producer of tungsten-metal powder. It will be noted that only a small micron range is embraced between finest and coarsest of these grades. Particle sizes up to about 500 microns can be made without difficulty, for example by hydrogen reduction of ammonium paratungstate.

Recently, ultrafine tungsten powders have been produced for internal work at the Union Carbide Nuclear Company. Particle sizes in this ultrafine powder, range from less than 0.02 to 5.0 microns with an average of 0.06 micron. These powders are spherical in shape, have a bulk density less than 1 g/cm^3, and are highly fluid.

Actually, the present tungsten producers are capable of providing tungsten powder with practically any average particle size from about 0.1 to 500 microns. In practice, however, the bulk of the powder being used for fabricable forms of the metal and for consumable-electrode stock has an average particle size in the range of 2 to 10 microns. Ordinarily, powder in this size range is manufactured (i.e., by appropriate control in the oxide-reduction stages) to these limits rather than preparing them by blending various sizes to obtain the desired average. This is done to avoid an unfavorable distribution with detrimental effects in subsequent pressing and sintering operations.

Purity

Table 1 lists the current purity specifications of various companies and organizations for tungsten powder according to end usage in powder-metallurgy or melted product. From the questionnaire responses, it is apparent that oxygen, nitrogen, carbon, aluminum, iron, molybdenum, nickel, and silicon are regarded as the impurity elements of greatest importance.

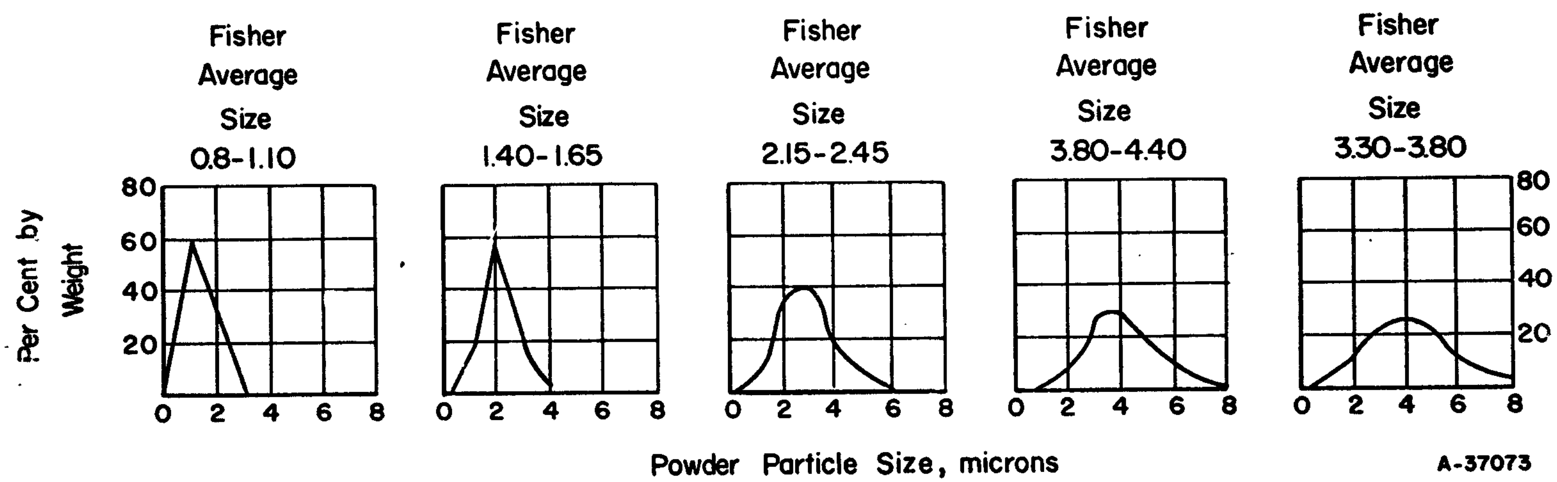


FIGURE 1. DISTRIBUTION OF PARTICLE SIZES IN SEVERAL GRADES OF PURE, COMMERCIAL TUNGSTEN POWDERS(4)

TABLE 1. CURRENT PURITY SPECIFICATIONS FOR TUNGSTEN POWDER

Company or Organization	Impurity Content, weight per cent: O	N	H	C	Al	Ca	Fe	Mo	Ni	Si	W
					For Use in Powder-Metallurgy Product						
Producers											
Cleveland Tungsten, Inc	0.20	--	--	0.01	--	--	0.01	0.005-0.030	--	0.01	--
Fansteel Metallurgical Company	0.10	0.005	0.005	0.02	0.005	--	0.02	0.02	0.02	--	--
Firth-Sterling Incorporated	0.30	--	--	0.01	0.01	0.01	0.02	0.01	0.02	0.01	--
General Electric Company	0.07	--	--	0.003	<0.001	--	0.002	0.003	<0.001	<0.001	--
Reduction and Refining Company	0.05	--	--	0.001	0.002	--	0.001	0.05	0.002	0.003	--
Sylvania Electric Products, Inc.	0.15	--	--	--	0.0005	--	0.002	0.005	0.001	0.001	--
Wah Chang Corporation	0.030	--	--	0.002	<0.002	--	0.003	0.05	<0.001	<0.003	--
Westinghouse Electric Corporation	--	--	--	--	0.005	--	0.005	0.002	0.002	0.01	--
Consumers											
Aerojet General Corporation (Sacramento)	0.02	0.02	0.01	0.025	--	--	0.05	0.015	--	0.05	--
Allison Division of General Motors Corporation	0.5	0.03	--	0.001	<0.001	--	<0.001	<0.003	<0.001	<0.001	--
General Electric Research Lab.	0.01	0.001	--	0.005	<0.001	--	<0.001	0.003	<0.001	<0.001	--
Raytheon Company	--	--	--	--	0.01	--	0.03	0.01	0.03	0.01	--
Universal-Cyclops Corporation	0.015	0.003	--	0.005	--	--	0.001	0.0025	0.001	0.001	--
					For Use in Melted Product						
General Electric Research Lab.	0.01	0.001	--	0.005	<0.001	--	<0.001	0.003	<0.001	<0.001	--
Oregon Metallurgical Corporation	0.03	--	--	0.01	--	--	--	--	--	--	99.9 min.
Universal-Cyclops Corporation	0.01	0.003	--	0.005	0.001	--	0.003	0.001	0.001	0.002	--
Wah Chang Corporation	0.010	--	--	0.002-0.010	<0.002	--	0.003	0.05	<0.001	<0.003	--
Westinghouse Electric Corporation (Blairsville)	--	--	--	--	0.002	--	0.002	0.01	0.002	0.002	--

The purity ranges specified for these eight elements are listed separately in Table 2. These figures show that oxygen is the largest single impurity in tungsten powder and that current specifications for oxygen as well as each of the other impurity elements vary widely, both for powder-metallurgical and melting applications.

TABLE 2. PURITY RANGES SPECIFIED FOR TUNGSTEN POWDER

Impurity Element	Impurity Content, weight per cent: For Use in Powder-Metallurgy Product	Impurity Content, weight per cent: For Use in Melted Product
O	0.05-0.5	0.01-0.03
N	0.001-0.03	0.001-0.003
H	0.005-0.01	--
C	0.001-0.02	0.005-0.01
Al	<0.001-0.01	<0.001-0.002
Ca	0.01	--
Fe	0.001-0.05	<0.001-0.003
Mo	0.0025-0.05	0.003-0.01
Ni	<0.001-0.03	<0.001-0.002
Si	<0.001-0.05	<0.001-0.002

The relatively high and extremely variable oxygen content of tungsten powder is, of course, due to the large surface area of powder and varies with particle size. For example, powder with an average particle distribution in the 2 to 4-micron range has a specific surface area of about 5000 cm^2/g while powder with an average particle size of 0.02 micron has a corresponding area of about 150,000 cm^2/g[5]. While no supporting data are available, it is known that tungsten powder can pick up appreciable quantities of oxygen on open-air storage. Consequently, where minimum oxygen content is desired, it is customary to store and ship the powder under a suitable dry inert gas (e.g., nitrogen, argon, etc.). Actually, for most current powder-metallurgy sheet applications, a high initial oxygen content of the powder is apparently of little concern since most of the gas is subsequently removed in sintering.

Two major reasons have been offered which partly account for the wide variance that presently exists with regard to purity specifications for tungsten powder, especially among consumers. These include a lack of knowledge of the effects of specific impurities on fabricability and mechanical properties and the difficulties associated with analyses of the metal. Impurity effects and analytical procedures are discussed in later sections of this report. It is sufficient to state here that, because of uncertainties in these areas, many consumers have not set any specifications for impurity elements in tungsten powder. Rather, these organizations either order to a minimum tungsten content (values ranging from 99.9 to 99.95 per cent) or, in some cases, simply request the highest purity powder available.

Prealloyed Powders

Doping Additions

Doping additions, generally, are proprietary mixtures of alkaline oxides with silica and/or alumina. The general purpose of these doping additions is to produce an interlocking elongated grain structure in the wrought and recrystallized metal. The overlapping structure reduces the tendency of offsetting at high temperatures by grain-boundary shear. In filament production, doping is identified with nonsag wire.

Little information is available on current doping procedures used by producers engaged in the consolidation of tungsten by powder-metallurgy procedures. Table 3[3] lists some of the mixtures known to have been used in the production of filament wire. In some instances thorium nitrate has also been added along with these dopes. Westinghouse Electric Corporation reports the use of the following doping agents as slurries: KCl (0.3 to 0.5 per cent), K_2SiO_3 (0.3 to 0.5 per cent), $AlCl_3$ (0.01 to 0.10 per cent).

TABLE 3. TYPICAL DOPES ADDED TO INFLUENCE RECRYSTALLIZATION IN TUNGSTEN WIRE[a]

0.45 per cent K_2O, 0.20 per cent SiO_2
0.35 per cent K_2O, 0.30 per cent SiO_2
0.15 per cent K_2O, 0.25 per cent Na_2O, 0.24 per cent SiO_2
0.15 per cent K_2O, 0.10 per cent SiO_2, 0.04 per cent Al_2O_3
0.15 per cent K_2O, 0.24 per cent SiO_2, 0.05 per cent Al_2O_3
0.145 per cent K_2O, 0.30 per cent SiO_2, + 0.025 per cent Al_2O_3 (the latter added to reduced powder)
0.15 per cent K_2O, 0.15 per cent SiO_2, and up to 0.1 per cent CaO

(a) Data are from Reference (3); amounts are expressed as percentage WO_3.

The foregoing modifying agents are normally added as solutions of soluble salts to the starting tungstic oxide. This makes possible the intimate mixing required to obtain an initially fine dispersion. Doping agents are not normally detectable in the final product due to the fact that these are practically all removed during sintering.

Thoria Additions

Thoria is commonly incorporated into tungsten by addition of thorium nitrate to tungstic oxide before reduction to powder. The purpose of the thoria is that of delaying recrystallization and grain growth. By such additions, the threshold temperature of recrystallization of tungsten may be increased, and the subsequent grain size restricted considerably.

The amount of thoria added normally ranges between 0.75 and 2.00 per cent. Additions beyond this range are not usually made because the slight increase in properties does not justify the additional difficulty in mechanically working such material.

Metallic Alloy Powders

At the present time, the only metallic "prealloyed" powders containing substantial amounts of tungsten which are available in commercial quantities are binary tungsten-molybdenum alloys. The production of these powders has been pioneered by Sylvania Electric Products who holds details of their preparation as proprietary information.

While the preparation of alloyed powders of practically any tungsten/molybdenum ratio desired appears practical, Sylvania's experiences to date with tungsten-rich compositions has been limited to powders of the 50W-50Mo and 85W-15Mo alloys. Particle sizes in the preparation of the prealloyed powders are controlled so that the resulting powders are described as having the same pressing and sintering characteristics of unalloyed tungsten.

Single Crystals

Within the past year, the Linde Company has introduced two single-crystal forms of tungsten that merit some mention as a new, high-purity source of the metal. Reportedly, production quantities of these single crystals are available either as spherical powders, in the size range of 5 to 150 microns, or as bars in diameters up to 3/4 inch and lengths to 12 inches.

Representative analyses of these crystals have been estimated(6) as follows:

Impurity Element	Content, ppm
Carbon	10
Hydrogen	1
Oxygen	25
Nitrogen	<20
Sulfur	10
Aluminum	100
Beryllium	100
Cadmium	100
Magnesium	1
Silicon	100

On the basis of the above analysis, these crystals offer no purity advantage over that which can be obtained with commercial powder after purification and consolidation by normal powder-metallurgical or arc-melting techniques.

CONSOLIDATION PRACTICES

Powder Metallurgy

Compacting

Methods. Traditionally, tungsten powders have been cold compacted by simple mechanical pressing in rectangular steel dies. This procedure is well adapted to making small shapes to be fabricated into rods and wire since swaging comprises the first stage of mechanical working. Small slabs intended for making sheet also can be made in this manner. Mechanical pressing is limited to shapes that can be adequately cold compacted by uniaxial compressive motion within a boxlike steel die. In order to achieve more uniform densification, a floating die may be used in which both top and bottom components move with respect to die walls.

A second cold-compacting method that has assumed increased importance within the last year or so is that of isostatic pressing. In this process tungsten powder is loaded into a plastic or rubber bag of appropriate size and geometry. The sealed bag is then placed in a confining chamber filled with a fluid medium and subjected to hydraulic pressures.

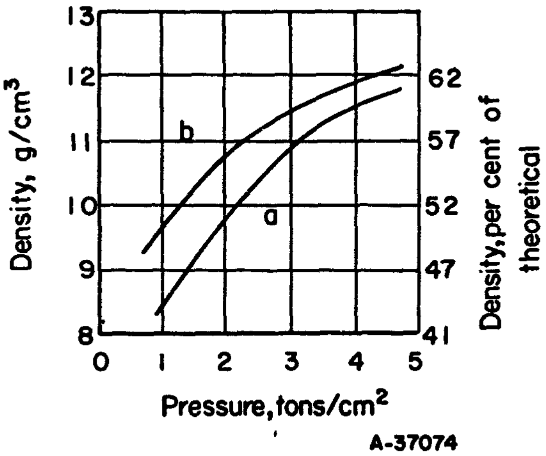
The geometry of the powder charge is retained as it is compacted from all directions, but dimensions are reduced according to the amount of compaction achieved. After compaction, the bag and compact are removed from the chamber. Following a stripping operation, the compact is ready for initial sintering.

Either of the above procedures can and has been adapted to the production of tungsten sheet bars.

Hot pressing is not as commonly used in consolidating pure tungsten powders as it is in the pressure sintering of WC-Co mixtures, or for certain tungsten alloys, for example. The temperatures at which the advantage of hot pressing becomes evident are those at which the pressed materials begin to exhibit marked plasticity. This temperature threshold for tungsten is well above the temperature range where steel dies have adequate strength, and, therefore, graphite molds are commonly used. At these higher temperatures mold contamination presents a problem. Therefore, hot pressing is not generally useful in producing a pure tungsten compact which is to be subsequently mechanically worked, such as sheet bar.

Pressures commonly used in cold compacting of tungsten powders vary for the most part from about 30,000 to 50,000 psi. The increment of improvement in densification drops off rapidly with increasing pressures above this range. One laboratory, however, reports the use of pressures as high as 80,000 psi for mechanically pressing small bar shapes, and a powder producer reports pressures as low as 10,000 psi for making isostatic pressings. For powder within the approximate range of 1 to 10 microns, compacting characteristics are sufficiently good that internal lubricants are usually not required.

Density. The details of the relationship between tungsten powders and cold-compacted densities are proprietary and were not disclosed by producers of powders. Maximum densities are achieved, however, where a range of particle sizes are involved and where powders are not too fine. Agte and Vacek[3] have shown (Figure 2) the advantage of a slightly coarser powder mixture in obtaining greater cold-pressed density. The curves reportedly continue to converge at higher compacting pressures.



Powder a: 0.8-μ avg diam
Powder b: 1.8-μ avg diam

FIGURE 2. INFLUENCE OF PRESSURE AND GRAIN SIZE OF METAL POWDER ON THE COLD-PRESSED DENSITY OF TUNGSTEN BARS[3]

The cold-pressed densities normally achieved for larger sized (isostatically pressed) compacts, range from extremes of about 50 to 75 per cent of theoretical. Densities in the 60 to 65 per cent range are most commonly attained.

Shapes and Sizes. A wide variety of cold-compacted shapes and sizes have been made from tungsten powders. These include rectangular and round bars, slabs, large cylinders, rings, tubes, rocket-nozzle-throat shapes, vanes, and other contoured sections. Maximum producible sizes reported in questionnaire answers reflected equipment limitations rather than technological limitations.

In general, shapes other than small bars and slabs are compacted in other than mechanical presses. Of primary interest in the production of sheet are rectangular sheet bars leading to directly workable bodies after sintering, and cylindrical or square shapes suitable for consumable electrodes. As noted earlier, compacting pressures in the range 30,000 to 50,000 psi are used yielding cold-compacted densities of about 60 to 65 per cent of theoretical.

Slabs usable as sheet bars after sintering, have been made in sizes up to 1-1/2 x 5 x 11 inches by mechanically pressing. Usually, however, the maximum slab sizes that can be made on present mechanical presses are somewhat smaller. For example, the maximum slab size producible at one facility is 1 x 4 x 8 inches and at another, 1-1/2 inches wide x 7 inches long. Another producer makes a maximum size of 1 x 3 x 30 inches. At present, tungsten sheet (in narrow widths) is made from rectangular bars which vary in sections from about 1 x 1 inch to 2 x 2 inches with lengths up to 2 to 3 feet.

Sheet bar slabs can also be made conveniently by isostatic pressing. Figure 3 illustrates a tungsten slab approximately 5-1/2 x 23 x 3/8 inch thick, made by this procedure.

It is of interest to note the consolidation practices outlined for evaluation by the Fansteel Metallurgical Corporation in their current powder-metallurgy tungsten sheet rolling program for the U. S. Navy(7). This work, being conducted on Bureau of Naval Weapons Contract No. NOw-60-0621-c, has, as its ultimate objective, the production of 3500 pounds of 0.060 x 18 x 48-inch high-quality tungsten sheet.

The initial phases of pilot powder evaluation are to be conducted on small billets (about 1-1/2 inches in diameter by 4 inches tall) isostatically pressed in gel molds. The follow-up work will be conducted on bars of 9-kilogram initial size, prepared both by mechanical and isostatic pressing, in 2-inch widths and 30-inch lengths.

Electrodes may be made either by mechanically pressing or by isostatic pressing. Square bars pressed mechanically are sometimes used for first melts in making small-diameter ingots. These ingots then may be used as electrodes for second melting. For electrodes to be used in making larger-diameter ingots, isostatic pressing is preferred.

So far as isostatic pressing of electrode sections is concerned, facilities already exist for the production of rounds in diameters up to 14 inches and lengths to 15 feet. At least one 10-inch-diameter tungsten billet, 4 feet in length and weighing 3000 pounds, has been produced by this process(8).

Sintering

Preliminary Considerations. Sintering serves the twofold purpose of purifying and consolidating compacted-tungsten-powder forms into shapes which have useful strength. Sintering is basically a diffusion process in which the sintering atmosphere, time, and temperature are the three variables of main importance. Of these, temperature has the greatest effect on the rate of consolidation in accordance with reaction-rate-theory concepts.

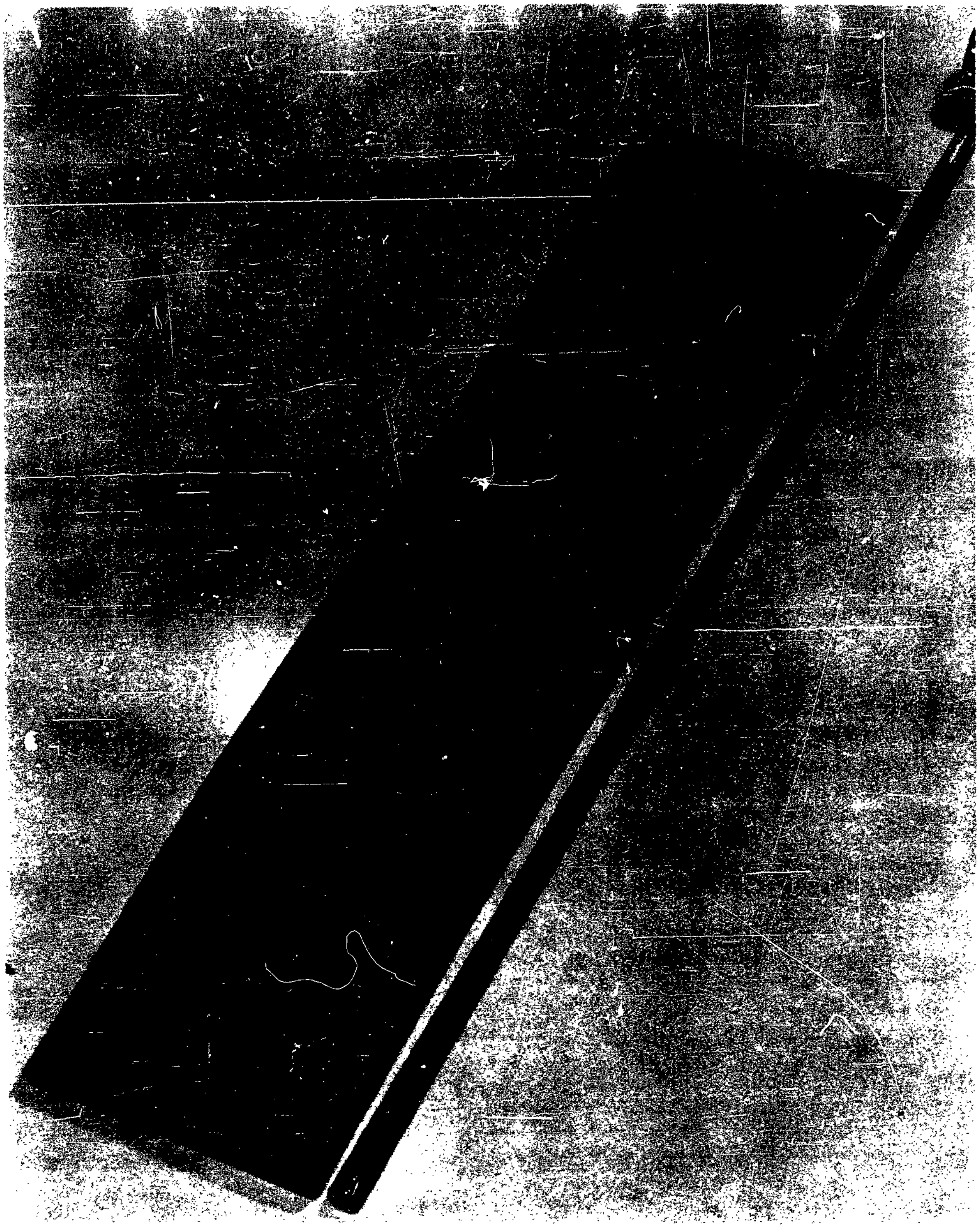


FIGURE 3. TUNGSTEN SHEET-BAR SLAB MADE BY ISOSTATIC PRESSING

Courtesy Sylvania Electric Products, Inc.

The influence of sintering temperature on densification of cold-compacted tungsten powders is illustrated by the curve of Figure 4[3]. In the work represented, sintering times of 1/2 hour were maintained during sintering at successively higher temperatures. The advantage of higher temperatures in producing a well-consolidated shape in a short time is evident.

Also of interest in this connection is recent work described by Pugh and Amra[9]. During 1/2 hour at 3100 C (5610 F) in vacuum, a density of 18.4 g/cm^3 (95.5 per cent of theoretical) was obtained. At 2820 C (5110 F), for the same time, the density achieved fell off to 17.4 g/cm^3 (90.0 per cent of theoretical), at 2540 C (4600 F), to 16.0 g/cm^3 (82.5 per cent of theoretical), and at 2300 C (4170 F), to 14.57 g/cm^3 (75.4 per cent of theoretical). These investigators found also that the apparent activation energy of densification increases as densification proceeds in qualitative agreement with the curve of Figure 4 just noted.

The effect of powder properties on sintering behavior has been discussed in some detail by Agte and Vacek[3]. Although finer powders compact less easily, they tend to sinter more readily. High purity also accelerates the consolidation rate. Powder particles should possess rough clefted surfaces for maximum sinterability. Figure 5 illustrates particle-size ranges for two experimental powders designated by Smithells and co-workers[10] as fine and coarse. The sinterability of these powders and those from other sources are illustrated in Figure 6. Curve B represents pure tungsten powder with optimum sintering properties. Allowance for the variation in sinterability of various tungsten powders is being taken into account in the current Fansteel tungsten sheet rolling program (Contract NOw-60-0621-c)[7].

The time-temperature combinations used vary widely and are chosen on the basis of obtaining the densification required for the operation to follow. In the case of larger sections where self-resistance heating cannot be employed, maximum sintering temperatures are limited by furnace equipment. Since time and temperature are mutually reinforcing in the densification process, such larger sections often require longer heating times. In effect, sintering time is a dependent variable with respect to the temperature provided by the available sintering facilities. Because of the chemically reactive nature of tungsten, hydrogen or vacuum are the most commonly used sintering atmospheres.

Pertinent information regarding the sintering experiences with consumable electrodes and compacts for direct working are summarized below:

Consumable Electrodes. Data for the sintering of tungsten electrodes for arc-melting purposes as obtained in the survey, are given in Table 4. The wide range of temperatures and times, and resultant densities indicates that each producer and/or fabricator has his own workable sintering procedure, the details of which are adjusted to his production facilities. For these reasons, valid generalizations in regard to "optimum" or "typical" schedules, are difficult to make.

Electrode densities as low as 50 per cent of theoretical have been reported, but these are certainly lower than those most commonly used.

Lower densities can be tolerated in arc-melting stock than for shapes where mechanical working is to follow. However, very low electrode densities are not

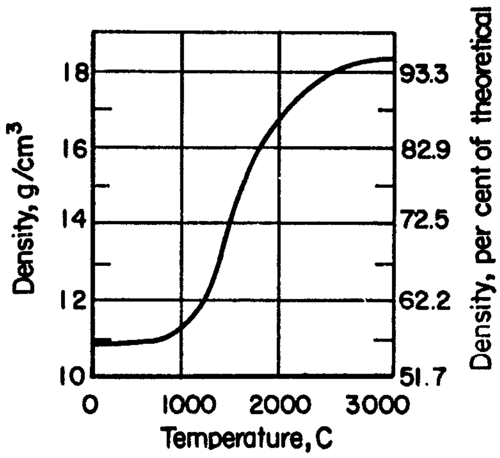


FIGURE 4. INFLUENCE OF SINTERING TEMPERATURE ON THE DENSITY OF PURE TUNGSTEN BARS(3)

Bars were 10 x 10 x 320 mm, sintered for 1/2 hour.

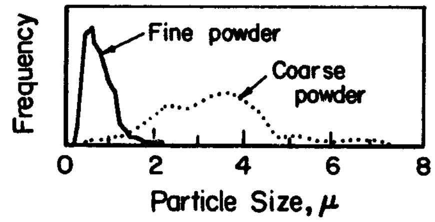
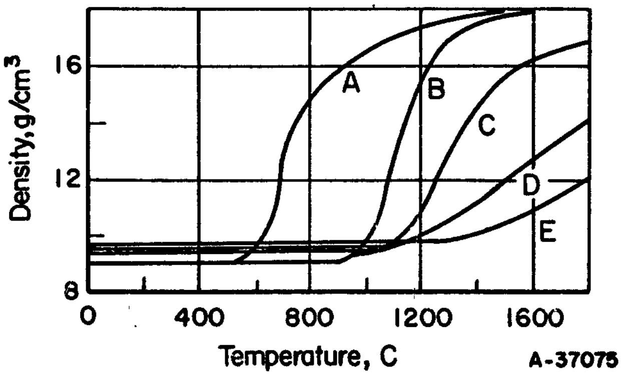


FIGURE 5. PARTICLE-SIZE DISTRIBUTION FOR FINE AND COARSE TUNGSTEN POWDERS(10)



A. Powder with good sinterability (10 per cent Ni addition)
B. Powder with good sinterability
C. Fine-grain powder
D. Coarse-grain powder
E. Powder with poor sinterability (with nonsag additives)

FIGURE 6. SINTERABILITY OF VARIOUS TUNGSTEN POWDERS(3)

TABLE 4. SINTERING OF COMPACTED POWDERS INTENDED FOR USE AS CONSUMABLE ELECTRODES

Organization	Reported Sintering Temperature or Range of Temperatures, F	Reported Sintering Time, hr	Sintering Atmosphere	Reported Density Range of Densities, per cent of theoretical
Fansteel Metallurgical Corp.	--	--	Dry H_2	90-92
Firth-Sterling Inc.	--	--	H_2 or vacuum	50-95
General Electric Corp. (Cleveland)	Above 3250	--	H_2	65-92 ± 3
NASA (Cleveland)	3000-3500	4-24	H_2 or vacuum	70-75
Sylvania Electric Products Inc.	3200-3350	--	--	65-95
Wah Chang Corporation (Albany)	2550	2	Vacuum (1-20 μ)	85-87
Westinghouse Electric Corporation	3100-3300	4-100	H_2	68-98

necessarily optimum. Noesen and Hughes have shown that melt-off rates are density-dependent, electrodes of lower densities yielding higher melt-off rates[11,12]. Rapid melt-off rates tend to decrease the rate of purification in the arc-melting process.

Compacts for Direct Working. Where self-resistance heating for sintering is required, the as-pressed compacts are presintered to permit subsequent handling. Temperatures in the range 950 to 1150 C (1750 to 2100 F) for periods of about 1/2 to 2 hours are employed. The customary atmosphere used for this purpose is dry hydrogen. Substantially no densification or purification is obtained in this type of presintering operation.

The minimum density desired for direct workability in sintered tungsten billets varies with the type of working operation. As discussed later, experiences indicate that the minimum varies from about 88 to 90 per cent of theoretical, for forging, rolling, and extrusion.

Densities of this order can be obtained in a relatively short time (1 to 2 hours) with relatively high (4400 to 4600 F) sintering temperatures. However, the only practical means of attaining these temperatures in commercial operations has been through the use of self-resistance heating. The present upper limit of section size which can be handled by self-resistance heating appears to be about 2 square inches. This is largely due to economic considerations which include power and power cost limitations as well as metal losses from the unsintered bar ends.

The producers' practices for sintering sheet bars of section sizes larger than about 2 square inches are not considered open information. However, the projected work outline of Fansteel[7] in the Navy sheet rolling program indicates that this organization has an induction-sintering furnace capable of heating 9-kilogram, 2 x 30-inch tungsten bars at temperatures to 4000 F. For evaluating a higher sintering temperature (4900 F), self-resistance heating is to be used.

As indicated by the information in Table 4, it is apparent that very few organizations have induction- or radiation-heating furnaces which are capable of sintering large tungsten sections at temperatures above 3300 F. It is also evident that appreciably long sintering times at temperatures in the 3000 to 3300 F range are required to obtain a densification of 88 per cent of theoretical or greater. It appears, therefore, that the present lack of furnaces capable of sintering large-size sheet bars at temperatures above 3500 F may place a serious economic disadvantage on the production of wide tungsten sheet by the powder-metallurgy process.

Some tungsten-base alloys are amenable to production by sintering mixtures of the pure powders. In producing tungsten-molybdenum alloys, the sintering cycle commonly employed for tungsten is sufficient to ensure the formation of a homogeneous alloy[1,3,13]. Tungsten-base tantalum or columbium alloys also can be made by starting with mixtures of tungsten and tantalum (or columbium) powders. Goetzel[13] notes that alloys containing not more than 5 per cent of either alloy are ductile while all other compositions are relatively brittle and difficult to work.

Purification Reactions. The use of vacuum in sintering has presently become more common as the result of efforts to produce a purer product. This same trend is reflected in hydrogen sintering in the use of drier hydrogen atmospheres. So far as is known, the quantitative advantage of vacuum sintering over hydrogen sintering, with respect to purification achieved, has not been measured. Allen, et al.[14], did not discover any advantage in the use of vacuum over hydrogen in the removal of residual oxygen. It is to be expected, however, that vacuum treatment would be decidedly advantageous in the removal of other impurities, e.g., metallic impurities in concentrations and at temperatures at which they were appreciably volatile.

The ease in removal of interstitial contaminants carbon, oxygen, and nitrogen from tungsten is believed to be proportional to their solubilities in tungsten, as noted by Allen, et al.[14]. The solid-solubility limits for interstitial contaminants in tungsten roughly determined by them are approximately as follows:

Element	Approximate Solubility, ppm, at Indicated Temperature
C	100-200 at 3600 F
O	30-40 at 3100 F
N	1-2 at 4200 F

The foregoing expectation was borne out by experimental work as shown by impurity levels at different stages of processing treatment. These results are as follows:

Processing Stage	Impurity Content, ppm C	O	N
As-received powder	20	310	--
Powder, after sintering for 2 hours at 4700 F in either vacuum <u>or</u> hydrogen	19	5	<4
After rolling sintered piece to 25-mil-thick sheet	54	50	<3
After 1-hour vacuum anneal of sheet at 4700 F	38	6	<0.4

Although oxygen could be readily removed from tungsten, apparently by volatilization as tungsten oxide, to levels below 10 ppm by vacuum or hydrogen annealing, carbon was not so easily eliminated. Apparently this was removable only by CO formation with residual oxygen.

Pugh and Amra[9] have reported the results of vacuum purification studies in which small rectangular bars 3/8 x 3/8 x 24 inches long were self-resistance sintered at temperatures between 3270 and 5610 F. The rate of densification was the primary objective of this work, but purification trends also were examined. It was found that the improvement in impurity content at higher sintering temperatures was remarkably good as illustrated in Table 5. The oxygen content of the bar sintered for 2 hours at 5610 F is comparable to that of the best arc-cast ingots.

TABLE 5. GAS ANALYSIS OF SINTERED TUNGSTEN[9]

Conditions of Vacuum Sintering[a]		Gas Content[b], ppm		
Temperature, F	Time, min	O_2	N_2	H_2
5610	120	2	1	0
5110	122	8	2	1
5110	30	12	2	1
4600	360	6	1	0
4600	120	5	1	1
3670	120	57	5	2

(a) Approximately 10^{-4} mm Hg.
(b) ±5 ppm.

It is reported that one European producer has used an alternating vacuum-hydrogen treatment, terminating in a vacuum-sintering stage, for consolidating tungsten powders. Goetzel[13] refers to German Patent No. 635,644 in connection with alternating hydrogen-vacuum sintering. The hydrogen for this purpose is dried to a low dew point. Wet hydrogen, as used by some facilities, leaves a small residue of oxygen in the tungsten.

Melting

There are several procedures in current use for melting tungsten. These include plasma-jet spraying, zone melting, levitation melting, vacuum arc melting, and electron-beam melting.

Of these processes, only the vacuum arc- and electron-beam-melting practices have shown a demonstrated capability for producing tungsten ingots large enough to obtain sheet in the sizes of interest in the current AMC program. Hence, only these two melting processes are reviewed in this report. For convenience, the vacuum-arc-melting experiences have been divided into two parts, separating those of consumable-electrode melting from those of skull melting*.

Consumable-Electrode Arc Melting

As early as 1913 it was demonstrated that sintered tungsten could be arc melted to produce small workable pieces of tungsten[1]. In 1951 the feasibility of producing small forgeable ingots of tungsten by arc melting was demonstrated at Battelle Memorial Institute[15]. Since this time a considerable amount of information of direct interest to the technology of melting in general has been generated. The status of some of this knowledge is summarized in References (16) through (26), inclusive.

*Consumable electrodes are used in both of the above categories. By "consumable-electrode melting" is meant melting into a cold-wall mold in contrast to "skull melting" in which the crucible is in effect a liner of solidified ingot material.

Because the consolidation of tungsten by arc melting is a specialized area in the broader field of vacuum arc-melting technology, much of the current practice for tungsten has been adapted from experience with other metals, notably the arc melt consolidation of molybdenum. Although an over-all similarity exists for all procedures, considerable differences in details of practice exist from facility to facility. At least ten different organizations have reported successful experiences in the preparation of sound tungsten and tungsten-alloy ingots in diameters up to 12 inches by consumable-electrode arc-melting techniques.

Electrode Configurations. A variety of electrode configurations are used, depending on the circumstances peculiar to each facility involved. For making small-diameter ingots, several organizations have used square-electrode sections with good results. The PSM (Press-Sinter-Melt) machine at Climax Molybdenum utilizes a hexagonal-section electrode. In almost all other instances, a simple round or cylindrical electrode section is used. Table 6 summarizes reported consumable-electrode melting practice. Electrode configurations used are noted in the second column.

Joining of Electrodes. Electrode joining procedures are reported in the third column of Table 6. In interviews at several arc-melting facilities, it was noted that successful joining of electrodes by inert-atmosphere arc welding was complicated by cracking at the weld. Cracking tendencies were more aggravated in larger electrode sections where the weld constituted a relatively small percentage of cross-section area.

Arc Characteristics. A characteristic of the vacuum arc is its very low voltage drop. The arc voltage is a dependent function of a number of variables including arc current, electrode separation, electrode material and arc-atmosphere composition and pressure.(27) In general, voltages are not proportional to ingot sizes. These facts are reflected in reported arc-melting voltages (Table 6, Column 6). For the most part, these were in the 25 to 40-volt range, with voltages for smaller ingots lying in the vicinity of 30 volts. In contrast, amperages showed a constant and appreciable increase with ingot size as illustrated in Figure 7.

It will be noted that d-c straight polarity is used at most melting facilities. With this polarity, the ingot pool is the anode which therefore receives the larger share of heat developed by the arc. As noted by Johnson(27), the arc-heat distribution shifts progressively away from the cathode and toward the anode as the cathode melting point increases. With the high melting point of tungsten this becomes an advantage from the standpoint of purification(11) and of maintenance of a wide molten pool. It has caused some trouble, however, especially in casting small ingots, due to crucible burnthrough. For example, in the preparation of 1-3/8-inch-diameter ingots at one facility(28), operation of a negative electrode caused considerable trouble by crucible perforation because of the large amount of power dissipated at the anode. On the other hand, when the consumable electrode was made the anode, ingot condition was poor. As a result a-c power was finally used to make these small tungsten ingots, which were intended for extrusion studies.

TABLE 6. SUMMARY OF DATA FOR CURRENT CONSUMABLE-ELECTRODE ARC-MELTING PRACTICE AS APPLIED TO TUNGSTEN

Organization	Electrode Configuration	Electrode Joining	Melting Conditions: Electrode Material	Ingot Diameter, in.	Voltage	Amperage	Polarity	Furnace Atmosphere
Climax Molybdenum Company	Round	None	W	2-5	--	--, ac	--	Vacuum
	Hexagonal	Resistance weld	W	5-9	--	--, ac	--	Vacuum
			85W-15Mo	12	--	--, ac	--	Vacuum
General Electric Company Research Lab	--	Thread and tap	W	4	40	5,000, dc	--	Vacuum (~2μ)
NASA (Cleveland)	Round	Arc weld	W	2-1/2	28-30	4,500-4,700, dc	Reverse	Vacuum
Oregon Metallurgical Corporation	Square	Heliarc weld	W	4	--	--	--	--
	Round		85W-15Mo	12	25	30,000, dc	Straight	Vacuum (~20μ)
Union Carbide Metals Company	Square	Arc weld	W	1-1/2	23-25	2,200, ac	--	Vacuum
	Round		W	2	25-26	3,500, ac	--	Vacuum
Universal-Cyclops Steel Corporation	Round	To 2 in. weld; >2 in., thread and tap	W	4-8	30-35	4,000-15,000 dc	Straight	Vacuum (~10μ)
			85W-15Mo	11-3/4	42	23,000, dc	Straight	Vacuum (~10μ)
U.S. Bureau of Mines Albany, Oregon	Square	Arc weld	W	2-1/2	30-32	3,500-4,000, dc	Straight	Vacuum (~150μ)
	Round		W	3	30-32	4,800-5,000, dc	Straight	Vacuum (~150μ)
			W	4	30-32	4,000-4,500, dc	Straight	Vacuum (~150μ)
			W	5	30-32	7,500-8,000, dc	Straight	Vacuum (~150μ)
Wah Chang Corporation	Square Round	Electron-beam or arc weld	W	3-1/2	24-30	6,000-6,500, dc	Straight	Vacuum
Westinghouse Electric Corporation (Blairsville)	Round	Thread and tap	W	4	30-32	5,500, dc	Straight	Vacuum (~10μ)
			W	6	40	7,000-10,000, dc	Straight	Vacuum (~10μ)

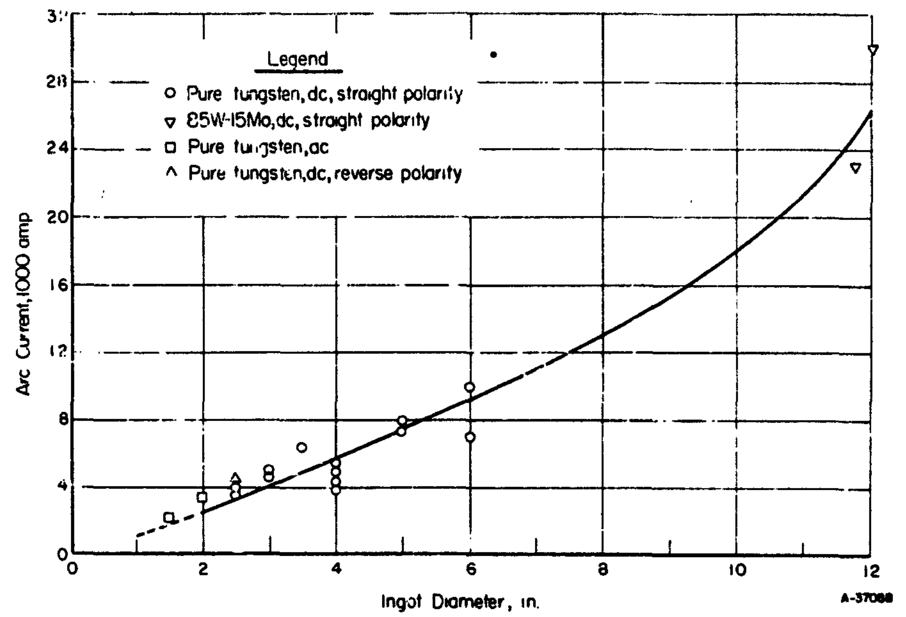


FIGURE 7. ARC CURRENT VERSUS INGOT DIAMETER IN VACUUM ARC MELTING TUNGSTEN AND TUNGSTEN ALLOYS

The melting atmosphere in all instances is vacuum, at various pressures. Some of the variation in reported melting pressures is no doubt due to differences in location of pressure-measuring devices within evacuated systems. In most instances, no estimate of pressure in the arc was available. One facility estimated pressures above the molten tungsten pool to be about 1 micron. Another estimate was as high as 4 mm. Noesen(11) has reviewed this topic. In hollow-electrode melting of molybdenum, with a furnace body pressure of 0.8 micron, a pressure of 3000 microns was measured at the upper end of the electrode.

Melt-off rate is an important factor in consumable-electrode arc melting of tungsten. Purification of the melt is a function of this rate, more rapid melting yielding appreciably less pure ingots. Among other factors, electrode density (and correspondingly, electrode resistivity) is of considerable importance in melt-off rate. It has been suggested earlier that low electrode densities are necessary to melt tungsten into small diameter molds (approximately 2-1/2 inches)(23). On the other hand a recent report from Westinghouse(29) showed that, during consumable-electrode melting of a 2-1/2-inch tungsten ingot, rapid sintering of the electrode occurred. In this work, initial density was 57 per cent of theoretical. After 30 seconds of melting the density of the unmelted electrode had increased to 72 per cent of theoretical. In the subsequent 90-second melt off of the same electrode, the density of the unmelted electrode was 89 per cent of theoretical. The conclusion reached in this study is that d-c straight polarity consumable-electrode arc melting of tungsten into small-diameter molds results in such rapid sintering that electrode densities are not important beyond the brief initial stage where sintering has not been effective in densifying any poorly consolidated material. It has been noted(30), however, that in arc melting of large sintered electrodes (3 to 4-inch diameters) with densities of about 70 per cent, warpage due to nonuniform shrinkage may result in burnthrough of the furnace wall.

Noesen(22) has shown that the addition of diatomic hydrogen to the furnace atmosphere lowers the electrode melt-off rate. Other factors of importance in melt-off rates are power input, amperage, arc polarity, electrode cross-section area, crucible cross-section area, and the presence or absence of gaseous elements which either change the voltage characteristics of the arc or redistribute the heat dissipation between anode and cathode surfaces(11).

In connection with electrode densities, Westinghouse reported(31) some experience with explosions of sintered electrodes which had previously been immersed in water. These electrodes (density 90 per cent of theoretical, 50W-50Mo alloy) blew apart on melting, even though one of the electrodes had subsequently been "baked out" at 1000 F before melting.

It is apparent from the foregoing discussion that optimum melting conditions vary from group to group depending on particular experiences within each organization. These experiences in turn have been modified considerably by objectives in melting and by equipment available to carry on the work. In many cases, the choice between a-c or d-c power, and between reverse or straight polarity was strongly conditioned by empirical observations, and by equipment limitations.

Ingot Sizes. The largest diameters of ingots reportedly made by consumable-electrode cold-mold arc melting are summarized in Table 7. Figure 8 illustrates a 3-inch-diameter ingot as arc melted from a 1-inch-diameter tungsten electrode. Figure 9 represents a 5-inch-diameter ingot as made by arc melting the previous 3-inch ingot as a consumable electrode. The electrode stub is also shown for comparison.

TABLE 7. FACILITIES AT WHICH TUNGSTEN AND/OR TUNGSTEN-BASE ALLOY INGOTS IN DIAMETERS OF 4 INCHES OR LARGER HAVE BEEN MELTED BY COLD-MOLD CONSUMABLE-ELECTRODE ARC MELTING

	Maximum Ingot Diameter, in.	
Facility	Unalloyed W	85W-15Mo
Climax Molybdenum Corp.	9	12
General Electric Corp.	4	--
Oregon Metallurgical Corp.	4	12
Universal-Cyclops Steel Corp.	8	12 x 70 in. long
. S. Bureau of Mines	5	--
Wah Chang Corporation	4-1/2	4
Westinghouse Electric Corp.	6	--

Ingot quality is of prime concern in consolidation by arc melting. An ingot that is poorly consolidated at wall surfaces will require excessive amounts of circumferential machining to produce a mechanically deformable shape. Table 8 illustrates some experiences reported in the recovery of sound ingot material. Individual figures reported are not necessarily typical because of the limited amount of experience of most melters and because total amounts of tungsten that must be removed to produce sound surfaces are not consistent from ingot to ingot. Nevertheless it is apparent that at the present

FIGURE 8. THREE-INCH-DIAMETER TUNGSTEN INGOT ARC MELTED FROM A ONE-INCH-DIAMETER TUNGSTEN ELECTRODE

Courtesy U.S. Bureau of Mines, Albany, Oregon.

FIGURE 9. FIVE-INCH-DIAMETER TUNGSTEN INGOT ARC MELTED FROM A THREE-INCH ELECTRODE (LEFT)

Courtesy U.S. Bureau of Mines, Albany, Oregon.

time approximately 1/2 inch of metal must be removed from the ingot radius for adequate cleanup, yielding about 70 per cent ingot recovery.

The macrostructure of unalloyed cold-mold arc-cast tungsten has the typically coarse appearance as illustrated in Figure 10. As noted by Noesen[11] impurities present on solidification tend to become highly concentrated at grain boundaries and are believed to act as a source of intergranular brittleness. Accidental fracture in arc-cast ingots tends to follow the boundaries shown.

Purification in Melting. Purification during arc melting of tungsten has been noted by a number of investigators. Some results reported by Noesen[11] are reproduced in Table 9. The marked decrease in interstitial impurity levels is self-evident. Data on the purification of tungsten by arc melting have also been given in an earlier paper by Noesen and Hughes[12]. In the work described, oxygen was reduced from an original electrode level of 100 ppm to less than 1 ppm, nitrogen from 10 ppm to less than 1 ppm, hydrogen from 5 ppm to less than 1 ppm, and carbon from 60 ppm to about 10 ppm. As noted by these authors, for the highest purity tungsten, melt-off rates must be kept to a minimum.

Morgan and Schottmiller[20] reported the data given in Table 10 comparing vacuum sintering and vacuum arc casting in the reduction of interstitials in tungsten. The tungsten powder used in this work was carbon reduced and of comparative low purity.

Typical analytical results achieved in the arc melting of carbon reduced tungsten powder also have been summarized by Morgan and Schottmiller[20]. The results are given in Table 11. These investigators found no improvement in ingot chemistry when hydrogen at reduced pressures constituted the melting atmosphere. The use of getters such as zirconium, manganese, and boron did not produce appreciable reductions of oxygen impurity levels.

Purification of tungsten during fusion is also of current interest in the welding and brazing of tungsten. In work undertaken at General Electric Company's Flight Propulsion Laboratory[32], a search is being made for additions which will purify tungsten and refine its cast structure during joining operations. The alloying additions to tungsten, selected for preliminary screening on the basis of their indicated abilities to promote purification by sub-oxide vaporization, to form stable compounds with interstitial impurities, or to promote grain refinement included the following (in per cent): 2.95 tantalum; 3.10 rhenium; 0.97 hafnium; 0.50 zirconium; 0.38 lanthanum; 0.24 yttrium; 0.03 boron; 0.033 carbon; 0.97 hafnium-0.030 boron; 0.97 hafnium-0.033 carbon; and 0.63 thorium. Tungsten with the above additions will be electron-beam melted to determine purification and grain-refining effects.

It is apparent on the basis of experience reported in the literature and of interviews with personnel engaged in melting, that analytical-chemistry methods will have to be improved before accurate assessment of the purification potentialities of the arc-melting process for tungsten can be made. It is also apparent that the effective purification during melting is dependent to a large extent on electrode purity level, and melting technique employed.

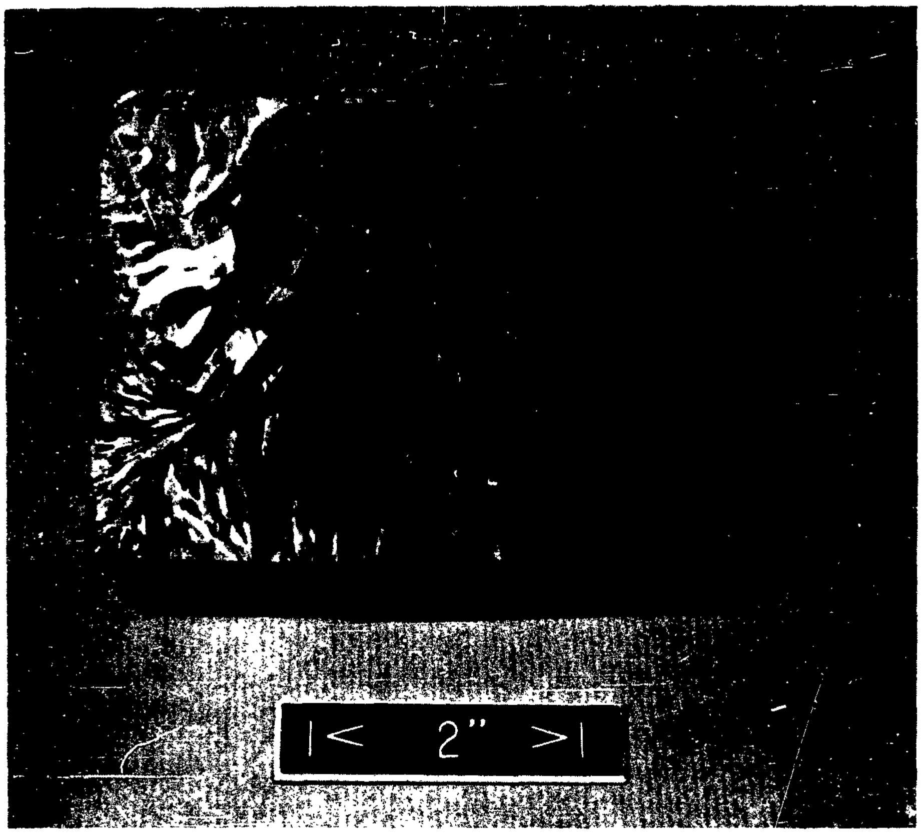


FIGURE 10. TYPICAL COARSE INGOT STRUCTURE IN ARC-CAST TUNGSTEN INGOTS

Courtesy U. S. Bureau of Mines, Albany, Oregon.

TABLE 8. REPORTED YIELDS OF SOUND INGOT RESULTING FROM SURFACE CLEANUP OF COLD-MOLD ARC-CAST MATERIALS

Organization	Arc-Cast Ingot Diameter, in.	Amount of Metal Machined from Radius, in.	Finished Ingot Diameter, in.	Per Cent of Original Ingot Recovered
A	--	1/2	--	--
B	4	1/4	3-1/2	~75
C	12[a]	1/2	11	~85
D	--	--	--	~50-60
E	1-1/2	--	--	~40-50
F	4	--	--	~60-70
G	4-1/2	3/8	3-3/4	~70
H	6	1/8	5-3/4	~92

(a) On 85W-15Mo alloy.

TABLE 9. PURIFICATION IN TUNGSTEN ACHIEVED IN A SINGLE PASS THROUGH A VACUUM-ARC-MELTING FURNACE[a](11)

Impurity Element	Impurity Content, ppm: Consumable Electrode	Impurity Content, ppm: Resultant Ingot
O_2	9	<1
N_2	3	<1
H_2	<1	<1
C	50	10

(a) Anode spot temperature: ~7500 F
Melting rate: 1.98 lb/min
Furnace pressure: 1μ.

TABLE 10. COMPARATIVE IMPURITY CONTENTS OF SINTERED[a] AND OF ARC-CAST TUNGSTEN FROM TWO LOTS OF POWDER[20]

Impurity Element	Raw Powder	Impurity Content, ppm: After Sintering in Vacuum (~0.1 μ Pressure), 1930 F, 5 Hr	3810 F, 4 Hr	After Vacuum Arc Casting
		Lot A		
Carbon	580	280	60	10
Oxygen	2200	1800	50	40
Nitrogen	50	24	11	5
Hydrogen	--	7	3	1
		Lot B		
Carbon	630	170	90	30
Oxygen	600	690	100	20
Nitrogen	40	17	4	1
Hydrogen	16	4	4	1

(a) 3/4-inch-thick compacts.

TABLE 11. TYPICAL PURIFICATION ACHIEVED IN VACUUM ARC MELTING OF CARBON-REDUCED TUNGSTEN POWDER[20]

Impurity Element	Impurity Content, ppm: Before Melting	After Melting
C	260	30
O	540	20
N	60	3
H	11	1
Si	70	26
S	120	10
P	<10	<10
Fe	450	40
Ni	30	5
Cu	4	2

Effects of Alloying Additions. A number of alloying additions to arc-cast tungsten ingots have been investigated. Common objectives have been an increase in elevated-temperature strength, improvement in high-temperature oxidation resistance, or the decrease of as-cast grain size in the ingot. The additions have been made in a number of ways, for example by incorporation of the alloying element or master alloy within the center of a hollow presintered tungsten electrode, by the original mixing of the alloying material with tungsten powder of the electrode, or by wrapping the electrode with a wire of the element to be added. Union Carbide Metals Co. has added titanium and columbium as master alloys and zirconium as sponge. In making graded-composition ingots, Climax Molybdenum Corporation has added alloying elements through an independent feeding procedure. Alloying additions made at this latter facility include molybdenum, tantalum, columbium, hafnium, zirconium, vanadium, iron, nickel, cobalt, silicon, and titanium[33,34].

Some difficulty has been experienced in the retention of the more volatile elements iron, cobalt, nickel, vanadium, silicon, and titanium. At Union Carbide, titanium could not be retained during vacuum arc melting in amounts above about 0.005 per cent. At Climax Molybdenum[33,34], iron, cobalt, nickel, vanadium, silicon, and titanium tended to be lost at temperatures and pressures prevailing in the melting zone. The volatilization of nickel and silicon during melting was essentially complete even at slightly above atmospheric pressures of argon gas in the melting chamber. Of 2.5 per cent cobalt added to a tungsten ingot, approximately 1.5 per cent was lost by volatilization.

Noesen[22] has noted the problem of alloying losses in making dilute tungsten-base alloys by arc melting. For example, vaporization losses of 65 per cent were encountered in the case of zirconium when added in small amounts.

Grain refinement in arc-cast ingots is of considerable importance. In this regard, the somewhat fortuitous effect of molybdenum has been noted with great interest by various organizations. This effect of molybdenum was probably first proven in large ingot sizes (9-inch diameter) by Climax who found[33] that molybdenum additions of as little as 10 per cent substantially decreased the as-cast grain size. This same effect apparently persists over the range of 5 to 70 per cent molybdenum.

Oregon Metallurgical Corporation has added small amounts of zirconium (about 0.2 per cent) in combination with 10 per cent molybdenum and 89.8 per cent tungsten and has reported a further reduction in the grain size of arc-melted button ingots. Of the original 0.2 per cent zirconium added, it has been estimated that only about 0.015 per cent remains in the cast ingot, the remainder having been lost by volatilization.

Significant grain refinement in tungsten has been also observed by Semchyshen and Barr[34] with binary additions of cobalt (up to 2.8 per cent cobalt) and vanadium (additions in excess of 2 per cent).

Grain refinement by the addition of boron has been noted by Morgan and Schottmiller[20]. Correspondingly, a large increase in hardness also was observed, but no appreciable amount of deoxidation occurred.

Boron additions to tungsten during arc melting have been tried at the Bureau of Mines, Albany Laboratories. No refinement was observed. However, the optimum boron addition may not have been made.

A grain-refining additive for arc-cast tungsten has been developed at General Electric Corporation's Refractory Metals Laboratory. According to this report, the as-cast grain size is considerably reduced and ingot workability has been correspondingly improved. Details are being withheld pending patent developments.

In this connection, U. S. Patent 1,179,009, "Method of Producing Malleable and Ductile Bodies of Tungsten or Tungsten Alloys" is of historical interest. This patent was granted to Alexander Just on April 11, 1916, who claimed the production of malleable tungsten by melting it with 2 per cent or less of boron nitride or other boron material.

In amounts above about 5 per cent, binary additions of tantalum or columbium to tungsten during arc melting have been found to have an embrittling effect. This has been the experience with these additions at NASA, Union Carbide Corporation(35), and Oregon Metallurgical Corporation. At the latter facility, inspection dye checks made on sections of 85W-15Mo-5Cb, 80W-10Ta-10Cb, and 80W-15Mo-5Cb ingots revealed extensive cracking. It is of interest in this connection that Goetzel(13) earlier noted the brittle behavior of tungsten-base alloys containing either columbium or tantalum in amounts exceeding 5 per cent.

Oregon Metallurgical has also experienced some difficulties with cracking of large ingot diameters of the binary 85W-15Mo alloy. These difficulties are alleviated by removing the ingots from the crucible while they are still hot and immediately transferring them to a furnace at 1650 F. After heating to 2800 F and holding for 4 hours, these are furnace cooled to 400 F.

Skull Melting

A variation of vacuum arc melting is that of skull melting in which the crucible is in effect a solid skull of the material being melted. The temperature gradient necessary to maintain such a skull is dependent on appropriate furnace design and operation. A sufficiently large pool of tungsten is maintained during electrode melt-off so that a substantial volume of metal can be poured into an adjacent mold by tilting the skull.

At the present time, skull-melting techniques for tungsten are in a less advanced state than are cold-mold arc-melting procedures. Groups which are currently most actively interested in skull melting and casting are those at Westinghouse Electric Corporation's Blairsville plant, the U. S. Bureau of Mines' Albany location, and the Oregon Metallurgical Corporation.

The work at Westinghouse Electric Corporation has been carried on in connection with a project on Manufacturing Development of Tungsten Alloys for Rocket Nozzles(36). Castings of 50W-50Mo, 70W-30Mo, 90W-10Mo, 99W-1Mo alloys and pure tungsten have been attempted. In melting 50W-50Mo alloy, both single and cluster electrodes were used. In the latter, 2-inch bars were assembled. Screw joints were employed. Approximately 100 kw per square inch cross section of electrode was required in melting the above alloy. Castings weighing up to about 70 pounds have been made. In subsequent work, in the skull melting and casting of pure tungsten, problems with contamination by the graphite mold material have been encountered.

Skull melting at the Bureau of Mines has been confined to small experimental heats. Figure 11 shows a tungsten skull remaining after pour-off of the molten tungsten pool. This illustrates the depth of melt obtainable in a skull-melting operation. Figure 12 shows a simple shape cast in a graphite mold.

At Oregon Metallurgical Corporation, skull melting is of considerable interest in connection with centrifugal casting. About 34,000 amperes at 35 to 40 volts is used to maintain an adequate volume of melt in the skull. Straight polarity is used to maintain as extensive a pool as possible. Castings weighing as much as 225 pounds have been made using 85W-15Mo alloy. Pure tungsten has not been skull cast. Centrifugally cast rings have been made in sizes up to about 6 or 8 inches wide by about 18 inches in diameter. The spinning mold operates with its axis of revolution in a vertical direction. The rough-cast ring is machined to clean it up and to produce parallel inner and outer wall surfaces.

A major advantage of this casting procedure is the production of fine chill-cast structures as illustrated in Figure 13. It is anticipated that the finer structure should be more amenable to mechanical breakdown.

Electron-Beam Melting

Currently, Stauffer Metal Company's Richmond plant is the principal facility engaged in electron-beam melting of tungsten, although other organizations have experimented with the process. The maximum size of unalloyed tungsten ingot that can be made at present is 4 inches in diameter. By the middle of 1961, Stauffer expects to produce 7 to 8-inch-diameter tungsten ingots several feet in length if necessary. For producing a 4-inch-diameter ingot approximately 15,000 volts dc are required with a corresponding current of 1 to 10 amperes. Furnaces are operated at about 10^{-2}-micron pressure. Estimated pressure above the melt is less than 1 micron. Present production capacity is 10,000 pounds of metal per month. It is expected that this will be doubled later on in the year.

"Critical composition control", through the use of proprietary additives, is used to reduce as-cast grain size in electron-beam melted tungsten. The results of such an addition is illustrated in Figure 14, showing the smaller grain size in the treated tungsten ingot. Comparison of the untreated ingot in Figure 14 with the arc-melted ingot structure shown earlier in Figure 10 indicates that electron-beam melting produces a comparatively larger as-cast grain size than that obtained in conventional arc melting.

The Wah Chang Corporation has also prepared unalloyed tungsten ingots, in diameters up to 4 inches, by electron-beam melting.

FIGURE 11. TUNGSTEN SKULL REMAINING IN SMALL EXPERIMENTAL FURNACE AFTER POURING

Courtesy U.S. Bureau of Mines, Albany, Oregon.

FIGURE 12. SIMPLE SHAPE OF TUNGSTEN AS CAST FROM SKULL MELT

Courtesy U.S. Bureau of Mines, Albany, Oregon.

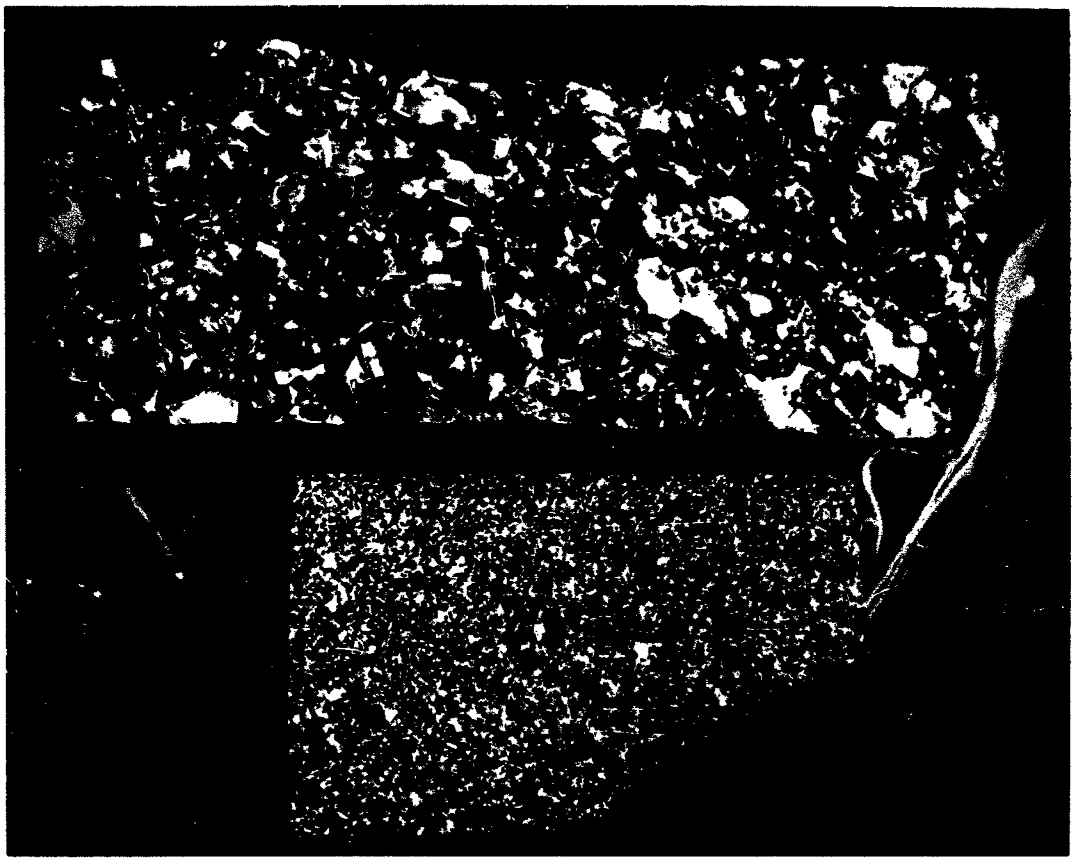


FIGURE 13. GRAIN SIZE OF 85W-15Mo ALLOY COLD-MOLD VACUUM ARC MELTED (ABOVE) AND SKULL MELTED AND CENTRIFUGALLY CAST (BELOW)

Courtesy of Oregon Metallurgical Corporation.

FIGURE 14. REDUCTION OF CAST GRAIN SIZE (LEFT) BY ADDING A PROPRIETARY GRAIN REFINER TO AN ELECTRON-BEAM-MELTED INGOT COMPARED WITH THE GRAIN SIZE OF AN UNTREATED ELECTRON-BEAM-MELTED INGOT (RIGHT)

Courtesy Stauffer Metals Corporation.

CONVERSION PRACTICES

All of the unalloyed tungsten sheet now being produced commercially is made by powder-metallurgical techniques. In this process, the sintered bar is rolled directly to finished product. Some producers elect to use an initial forging step prior to rolling.

Good success has been achieved in the forging and/or extrusion of large sintered and arc-cast tungsten shapes although relatively little effort has been made to convert the forged or extruded products into sheet. Nevertheless, unalloyed tungsten sheet, in commercial sizes, has been made from arc-cast ingot using extrusion and forging as intermediate working stages.

The same conversion practices are being used, with varying degrees of success, for a few tungsten alloys. However, the only alloys which have been made in commercial tungsten sheet sizes are those which contain doping or thoria additions. These have only been made by the powder-metallurgical process.

Swaging has been disregarded as a conversion practice of interest in the production of wide tungsten sheet even though this method of fabrication plays a vital role in the production of narrow sheet, rod, and filament wire. With the adoption of larger size equipment it is conceivable that swaging might become important in wide-sheet production.

Powder-Metallurgy Compacts

As noted above, powder-metallurgical techniques are the only ones being used at present to provide commercial tungsten sheet. Unfortunately, most of the details of commercial practice are regarded as proprietary. Hence, it is not possible to present an accurate step-by-step description of the techniques being used although some of the current limitations of commercial practices are apparent.

The widest commercial tungsten and tungsten alloy sheets are being made from sintered rectangular sheet bar rather than from rounds. While the feasibility of converting sintered rounds to sheet bar by forging or extrusion and forging has been demonstrated, neither of these processes has thus far been actually used commercially to produce sheet material.

Commercial Sheet Rolling

Unalloyed Tungsten. Until recently, tungsten sheet was not generally available from U. S. sources in widths over about 7 inches. As a result of the demand for larger sheet, primarily for fabrication into rocket nozzles, the producers are steadily improving their size capabilities. Thus, as shown in Table 12, sheet is now available in widths from 10 to 17 inches at thicknesses from 0.020 to 0.060 inch.

TABLE 12. CURRENT U. S. PRODUCTION CAPABILITY FOR TUNGSTEN SHEET BY MEANS OF POWDER-METALLURGICAL TECHNIQUES

Sheet Thickness, in.	Available Widths and Lengths of Tungsten Sheet, in.							
	Producer A		Producer B		Producer C		Producer D	
	Width	Length	Width	Length	Width	Length	Width	Length[a]
<0.020	--	--	3	Coil	--	--	2-3	120-300
0.020	10	24	4	60-72	--	--	2-3	120-300
0.040	17	17	6	36	--	--	2-3	180
0.060	15	15	7	20	10	36	2-3	120
>0.060	--	--	10	12	--	--	4	96

(a) Capability expected by June, 1961.

Historically, tungsten strip for the electronics industry has been produced from mechanically pressed and resistance-sintered sheet bars ranging in section sizes from 1 to 2 square inches and in lengths from 24 to 30 inches. This size of sheet bar represents about the maximum which can be economically sintered by self-resistance heating. All of the producers are now engaged in the pilot production of larger size sheet bars. While the specific sizes of these vary, their initial thickness is usually 1 inch or less. Surface areas range from 25 to 50 square inches with typical width-by-length dimensions being 3 x 12, 4 x 6, and 4 x 10 inches. With the development of suitable isostatic pressing techniques, the production of much larger sheet-bar sizes is now feasible (Figure 3). However, due to the general lack of suitable furnace sintering equipment, sheet production from bars of sectional areas exceeding about 4 square inches is not yet a routine commercial practice.

The target densities for as-sintered sheet bars range from 88 to 95 per cent of theoretical for four of the major producers. It is of interest to note at least one producer believes that specifying a maximum density (96 per cent) is of almost equal importance as specifying a minimum. In this case, densities above 96 per cent were viewed as undesirable in that higher working temperatures (i.e., approaching true hot-working conditions for tungsten) were necessary.

In the production of narrow strip from resistance-sintered sheet bar, Smithells[1] and Goetzel[13] have pointed out the need and desirability of preceding the rolling operation by forging or swaging. Here, a recommended sequence is to initiate the forging at 2730 to 3300 F with pneumatic hammers until a reduction of about 20 per cent in thickness is achieved. The use of such a forging operation in the breakdown of large sintered sheet bars is not an admitted practice by any of the producers although this procedure is under study. Fansteel, for example, has incorporated press forging as a processing variable for sintered sheet bar in the pilot evaluation stage of their sheet-rolling program for the Navy[7].

The available producer data for conditions of initial breakdown of sintered sheet bar are summarized in Table 13. Generally, preheating temperatures range from

2680 to 2910 F and hydrogen appears universally used as the preheating atmosphere. The available rolling-schedule data (Table 14) indicate reductions of 10 to 25 per cent are used in initial breakdown rolling with lesser reductions and lower temperatures being used in the intermediate and final rolling stages.

TABLE 13. CONDITIONS FOR INITIAL BREAKDOWN OF SINTERED TUNGSTEN SHEET BAR

Producer	Size of Sheet Bar, in.	Preheating Temperature, F	Preheating Atmosphere
1	1 x 3 x 12	2680	Hydrogen
2	1 x 4 x 6	Proprietary	Hydrogen
3	1 x 4 x 12	2730-2910	Hydrogen
4	12 x 12(a)	2910	Hydrogen

(a) Available by June, 1961.

Several producers admitted the desirability of getting the maximum reduction possible in the first rolling pass and one described plans for attempting to implement a 50 per cent single-pass reduction. It is of interest to note that these schedules (Tables 13 and 14) fit in well with the reduction schedules planned for evaluation by Fansteel in their tungsten-sheet-rolling program for the Navy (Table 15).

Goetzel[13] has pointed out that rolling of narrow strip is begun (after an initial forging reduction at 2730 to 3300 F) at 2370 to 2550 F. The successive rolling temperatures are decreased as the total reduction increases. The sheets are reheated in hydrogen between passes. When the sheet reaches about 0.040-inch thickness, it can be rolled at 390 to 750 F without appreciable oxidation. When thicknesses of 0.010 to 0.008 inch are obtained, heating of strip is no longer necessary and cold rolling may be employed. Intermediate heating facilitates the reduction, but the temperatures used must not reach those required for recrystallization (i.e., about 1830 F maximum).

Generally, oxidation during heating makes it difficult to produce sheet thinner than about 0.004 inch[13]. A method of producing considerably thinner (0.0008 to 0.0012 inch) sheet by pack rolling multiple sheets has been described[37]. Also, Schwarzkopf has described[38] a method of pack rolling tungsten between oxide- or asbestos-covered iron sheets, closed at the ends to exclude air. This technique has reportedly produced tungsten foils 3 to 5 microns thick.

Generally, no protective atmospheres (other than hydrogen) and no rolling lubricants are used in commercial rolling practice. As indicated in Table 16, surface-conditioning practices for the sheet in intermediate and final rolling vary widely. The industry has, as yet, not been able to eliminate difficulties with laminations, surface imperfections, and 45-degree cracking, especially in the wider sheet sizes.

At present, the widest sheet (at 0.020-inch thickness) which could be rolled at any of the producers' plants is 24 inches (one producer). The other producers are restricted to widths of 12 inches or less. The opinions of the producers themselves are

TABLE 14. REDUCTION SCHEDULES FOR ROLLING SINTERED TUNGSTEN SHEET BAR

Producer	Preheating Temperature, F	Reduction Per Pass	Reduction Between Anneals, per cent	Annealing Conditions Temperature, F	Annealing Conditions Atmosphere
1	2730-2910	25 per cent until ~0.1 in. thick	50	2640	H_2
	2730-2910	0.015 in. from 0.1-0.05 in. thick	50	2550	H_2
	2640-2820	0.010 in. from 0.05-0.02 in. thick	50	2460	H_2
2	2680-1830	10-15 per cent	Proprietary	Proprietary	H_2

TABLE 15. REDUCTION SCHEDULE FOR SINTERED UNALLOYED TUNGSTEN PLANNED FOR EVALUATION BY FANSTEEL ON NAVY CONTRACT NOw-60-0621-c[7]

Rolling Stage	Temperature Range, F	Reduction per Pass, per cent	Total Reduction, per cent	Final Sheet Thickness, in.
Breakdown	2730-3000	15-24	56	0.310
Intermediate	2380-2910	10-23	63-79	0.150
Final	2380-2910	10-21	81-91	0.060

TABLE 16. SURFACE-CONDITIONING PRACTICES FOR UNALLOYED TUNGSTEN SHEET

Producer	Processing Stage	Conditioning Treatment Used		
		Pickling	Grinding	Other
1	Intermediate	NaOH	No	--
	Final	$HNO_3 + H_2SO_4$	No	--
2	Intermediate	--	--	Sand blast
	Final	Yes	--	NaOH solution
3	Intermediate	Yes	--	--
	Final	Yes	Yes	--
4	Intermediate	Yes	Yes	Yes
	Final	Yes	Yes	Yes

at variance as to the limitations imposed by present mill equipment in rolling wide, thin-gage tungsten sheet. However, the consensus is that existing steel mills will be adequate for rolling tungsten sheet in widths of 24 to 36 inches at gages down to 0.020 inch. The most severe equipment limitation is the lack of adjacent heating furnaces which are (1) capable of providing a suitable protective atmosphere at temperatures to 3000 F, and (2) large enough in size to accommodate sheet in widths above 24 inches.

It is of interest to note the progress which has been made in the rolling of tungsten sheet at Metallwerke Plansee, the only heavy-equipment plant for refractory metals in Europe. Tungsten blocks, in sizes to 200 kg (440 pounds) are produced by pressing in a 3000-ton, hydraulic, single-action press at 20 tsi. The 200-kg-size block is cut into smaller pieces ranging in size up to 50 kg (110 pounds). These are sintered in a bell furnace by a proprietary method at temperatures around 4530 F. After sintering, the bars are rolled from a hydrogen furnace at 2910 F, with the rolling temperature being gradually reduced to about 1830 F. Intermediate stress-relief annealing at 2280 F is used roughly after every 50 per cent reduction. The finished tungsten sheet is not normally stress relieved unless the customer requests it or unless the sheet thickness is over 1/2 inch. Some pack rolling is used, particularly at lower temperatures, where a typical pack consists of Mo:W:W:Mo. Iron is not used for interleaving sheets[39].

Sheet production at Plansee has been limited to widths of about 18 inches maximum, the limit of their four-high, 60-cm (23-1/2-inch) mill. They are building a 120-cm (47-inch) mill which should be capable of producing 40-inch-wide sheet. In preliminary work at Deutsche Edelstahlwerke, sintered 18-kg (40 pound) sheet bars have been rolled (without the use of packs) with equipment intended for alloy sheets. Sheet sizes of 40 inches wide by 50 inches long have been made in gages down to 0.020 inch[39].

<u>Tungsten Alloys</u>. The state of the art for rolling powder-metallurgy sheet alloys of tungsten is considerably less advanced than for rolling unalloyed tungsten. The reasons for this are twofold. First, there has been no demand for large alloy sheet. Thus, the only nonmilitary market has been the electronics industry whose needs have largely been satisfied with narrow strip (about 2 inches maximum width) of thoriated or doped grades of the metal. Second, the preparation of alloy sheet is more difficult than the unalloyed tungsten.

Only four alloys of tungsten are available commercially in sheet form. These are the 1 and 2 per cent thoria alloys, available from several manufacturers, and two doped grades, General Electric's Type 218 and Sylvania's Type K-100. While the compositions of both doped alloys are proprietary, the 218 alloy was developed by General Electric several years ago primarily for application as a nonsag filament-wire material. Sylvania's K-100 alloy, on the other hand, is a relatively new material which was described[40] as a "lightly doped composition developed specifically for compaction into massive shapes". At the present time, neither of the thoriated grades nor the 218 alloy are available in widths above about 4 inches. The K-100 alloy, by comparison, has been rolled in widths of 7 to 10 inches, with lengths of 27 to 36 inches, at gages of 0.060 to 0.065 inch (Figure 15).

All of the processing data used in the manufacture of these sheet alloys are regarded as proprietary. However, the general reasons associated with the difficulties in preparing these are known and can be summarized as follows.

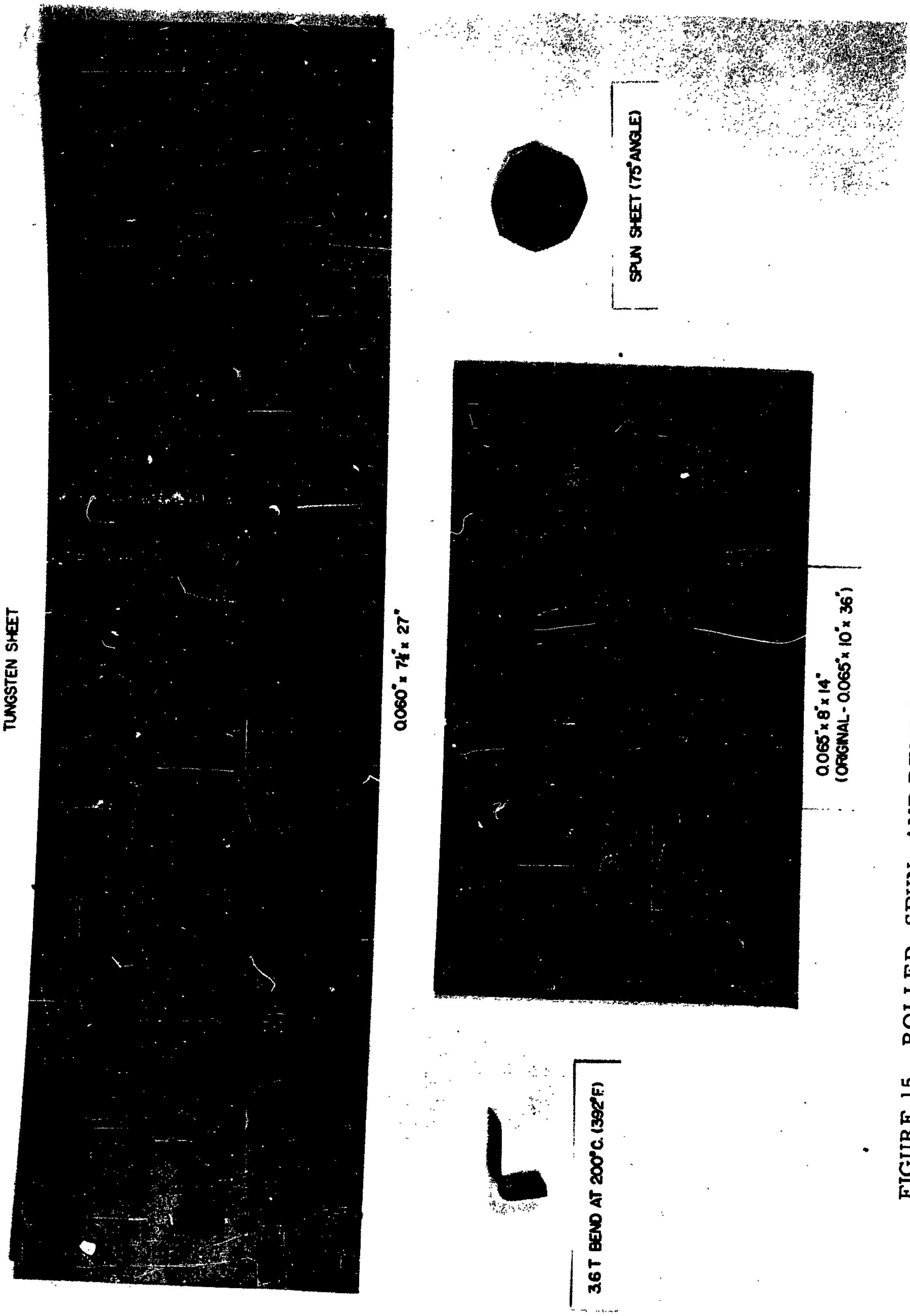


FIGURE 15. ROLLED, SPUN, AND BENT SAMPLES OF TYPE K-100 DOPED TUNGSTEN

Courtesy Sylvania Electric Products, Inc.

First, both thoria and the usual doping additions inhibit the densification of tungsten in sintering such that higher temperatures or longer times are required compared to those for unalloyed tungsten.

Second, thoria additions increase the deformation resistance of tungsten, at least in the range of working temperatures normally employed (i.e., 3000 F and below). This has generally been acknowledged by all producers of thoriated tungsten sheet. Hence, higher preheating temperatures are needed in the fabrication of the thoriated metal. This was shown in recent work by Westinghouse[41] in which the preparation of tungsten rod and sheet, containing additions of 2, 4, and 5 per cent thoria, was attempted.

In this work, the alloy bars (initially 3/4-inch square) were swaged to an 80 per cent reduction, starting at 2910 to 3000 F. Workability for the 4 and 5 per cent ThO_2 alloys was poor, giving yields of only about 25 per cent compared with a 50 per cent yield on the 2 per cent ThO_2 alloy. Sintered pieces of the 4 and 5 per cent ThO_2 alloys were successfully forged to a 50 per cent reduction in height at 3090 F. However, subsequent attempts to flat roll these forged pieces at 2910 F were not successful.

Experimental and Developmental

Comparatively little work has been reported on the development of wrought tungsten-alloy sheet made by powder-metallurgical practices. A considerable effort has been given to the extrusion and forging of sintered shapes of tungsten and a few of its alloys. All of this work, however, has been directly concerned with either the production of rocket nozzles and jetavators or simply the consolidation of bar stock for mechanical-property evaluations. Nevertheless, because these consolidation techniques may ultimately be useful in the production of sintered sheet bar, recent progress in these practices is described briefly here.

Direct Rolling. Both Westinghouse[41] and Battelle[14] recently completed research programs, under Air Force sponsorship, which dealt in part with the rolling of sintered tungsten alloys containing inert dispersed phases and/or doping additions. This portion of the Westinghouse work was concerned solely with thoria additions of 2 to 5 per cent and has been reviewed in the preceding section of this report.

The Battelle work was concerned primarily with a study of the effects of various additions on the workability, recrystallization behavior, and bend transition temperature of tungsten strip. The program was conducted using sintered alloy bars of 1/4 x 1/2 x 7 inches which were fabricated to 25-mil-thick strip as follows:

(1) Breakdown roll to 30 per cent reduction at 3270 F

(2) Consolidation resinter 5 minutes at 4710 F

(3) Roll to 30 per cent reduction at 3270 F

(4) Anneal 1 hour at 3270 F

(5) Roll to 75 per cent reduction at 2190 F.

Table 17 lists the compositions of the bars studied and the general results obtained. The pertinent observations from this work concerning the effects of the alloying additions on workability are summarized below:

(1) At additions of 2 volume per cent, dispersoids which are essentially thermodynamically stable in tungsten (e.g., ThO_2, ZrO_2, and HfO_2) have little detrimental effect on workability.

(2) Factors which reduce fabricability include (a) the presence of volatile additions (e.g., MgO and SiO_2) requiring sintering temperatures above 4700 F for adequate densification, (b) additions of dispersoids over 1 micron in size, and (c) additions of hafnium, tantalum, or zirconium in the form of nitrides or carbides. The latter compounds are essentially unstable (on sintering and processing at 3270 to 4710 F) and tend to decompose with resultant solid-solution strengthening of the tungsten matrix by the metallic component and a decrease in hot plasticity.

Extrusion. Four organizations have reported their experiences in the extrusion of sintered tungsten and tungsten-alloy billets.

The most extensive work in this area appears to have been that carried out at the Wright Air Development Division Refractory Metal Working Facility. One of the most outstanding recent achievements of this group has been the development[42] of an alumina-coated die which has been used successfully at temperatures from 1800 to 3400 F on a variety of sintered and arc-cast tungsten and molybdenum alloys. Whereas previous efforts with high-speed steel dies have given as much as a 1/4-inch washout in one 2800 F extrusion, the alumina-coated die has washed as little as 0.002 inch under the same condition. Alumina-coated dies have withstood as many as six high-temperature extrusions after which the coating has been removed, replaced, and the die used again for another series of extrusions.

Table 18 lists the extrusion data obtained by WADD on several sintered unalloyed and alloyed compacts for General Electric (Lamp and Metal Components Division). All extrusions were carried out with glass lubricants.

The accumulated experiences at WADD indicate that sintered unalloyed tungsten billets with densities of 93 per cent of theoretical or greater can be successfully extruded using the same extrusion parameters as for arc-cast tungsten. For sintered billets of lower density, more severe conditions (i.e., higher temperatures and greater reductions) are required.

The 1 per cent ThO_2 alloy (listed in Table 18) which failed to extrude successfully had a density less than 90 per cent of theoretical; this billet was later replaced with one of 96 per cent of theoretical density which has since been extruded 6/1 to round at 3400 F to a bar with "little roughness and no nose bursting"[44].

Metallographic examination of the unalloyed tungsten extrusions showed that the fine-grained structure, characteristic of sintered material, had been retained[43].

TABLE 17. ROLLING BEHAVIOR OF POWDER-METALLURGY TUNGSTEN-BASE DISPERSOID ALLOYS[14]

Intended Alloy Content, weight per cent	Type of Addition	Sintering Conditions: Temperature, C	Sintering Conditions: Time, hr	Sintering Conditions: Atmosphere	Sintered Density, per cent of theoretical	Fabricability[a]
Unalloyed W	--	2600	2	H_2	94.6	Good
	--	2600	2	Vacuum	90-94	Good
1 ThO_2	3-μ ThO_2	2600	2	Vacuum	91.8	Good
1 ThO_2	Aqueous	2600	2	Vacuum	94.5	Good
1 ThO_2, 0.2 Na_2O	Aqueous	2600	2	Vacuum	92.3	Good
1 ThO_2, 0.2 Na_2O	Aqueous	2600	2	H_2	91-92	Good
0.12 ZrO_2	0.01-μ ZrO_2	2800	2	Vacuum	94.5	Good
0.60 ZrO_2	0.01-μ ZrO_2	2600	2	Vacuum	94.8	Good
0.60 ZrO_2	1.9-μ ZrO_2	2600	2	Vacuum	93.6	Good
0.60 ZrO_2	Aqueous	2600	2	Vacuum	91.8	Good
3.2 ZrO_2	0.01-μ ZrO_2	2600	4	Vacuum	98.8	Good
0.25 SiO_2	0.02-μ SiO_2	2800	2	Vacuum	94.8	Good
0.39 MgO	2.3-μ MgO	2850	2	Vacuum	90.0	Fair
1 HfO_2	1.3-μ HfO_2	2800	2	Vacuum	93.5	Good
1 HfO_2	5.7-μ HfO_2	2800	2	Vacuum	86.6	Poor
0.41 Al_2O_3	0.03-μ Al_2O_3	2600	2	Vacuum	92.0	Fair
1.1 UO_2	1.2-μ UO_2	2600	2	Vacuum	93.0	Good
0.56 TiN	4-μ TiN	2800	2	Vacuum	93.3	Good
0.75 ZrN	2.1-μ ZrN	2800	2	Vacuum	95.4	Fair
1.3 HfN	2.6-μ HfN	2600	2	Vacuum	91.8	Poor
1.7 TaN	4.3-μ TaN	2600	2	Vacuum	92.0	Good
1.3 HfC	2.9-μ HfC	2600	2	Vacuum	94.7	Poor
1.5 TaC	4.5-μ TaC	2600	2	Vacuum	84.4	Fair
1.5 TaC	4.5-μ TaC	2800	2	Vacuum	92.6	Poor
1.4 Ta_2C	1.5-μ Ta_2C	2800	2	Vacuum	92.0	Poor

(a) Good = usable 25-mil-thick strip with negligible splitting or end cracking
Fair = usable strip with some edge cracking
Poor = alligatored and/or broken up.

TABLE 18. EXTRUSION DATA FOR SINTERED TUNGSTEN AND TUNGSTEN-MOLYBDENUM BILLETS[(a)] (42, 43)

WADD Facility, 3 x 6-Inch Billet Size

Composition, weight per cent	Reduction Ratio	Temperature, F	Required Force, tons	Extrusion Speed, ips	Results
Unalloyed W	4/1	2800	686	11.9	No visible cracks
	4/1	3000	650	8.2	5 per cent extruded
	4/1	3000	548	3.7/2	Excellent
	4/1	3000	598	3.7/2	Excellent
	4/1	3000	549	--	Excellent, smooth[(a)]
	4/1	3000	487	--	Excellent, smooth[(a)]
W-1 ThO_2	4/1	3000	550	--	Cracked
50W-50Mo	4/1	2600	640	13.8	5 per cent extruded
	4/1	3000	571	11.8	Rough
	4/1	3000	578	11.6	Nose smooth, back half rough

(a) Extruded using alumina-coated dies.

NASA has shown[45] that sintered tungsten billets of 2-1/4-inch diameter, clad with Type 304 stainless steel, could be successfully extruded at 2300 F with reductions of 4/1 and 5.35/1. A third clad billet stalled when a reduction ratio of 5.5/1 was attempted. Several arc-cast billets, extruded under similar conditions, showed rougher surfaces (ascribed to pull-out of the tungsten grains into the stainless steel), but less of a "dog-bone" effect.

High-velocity extrusion data were also obtained on sintered and arc-cast tungsten billets by NASA under the conditions shown in Table 19. "Although these data indicate that arc-cast tungsten extruded more successfully than sintered tungsten, the extruder (Dynapak Convair Division) reported no difference in ease of extrusion"[45]. The surface quality and soundness of these extrusions were generally good. Complete recrystallization, indicative of true hot working, was observed in the bar extruded at 3800 F with 45/1 reductions. Varying degrees of recrystallization were noted in the other bars extruded at lower temperatures and reduction ratios.

TABLE 19. HIGH-VELOCITY EXTRUSION DATA FOR UNALLOYED TUNGSTEN

NASA, 1 x 1-Inch Billet Size[45]

Reduction	Temperature, F	Dimensions of Extrusion, in. Diameter	Length
		Vacuum-Sintered Billets	
8.3/1	3500	Did not extrude	
9.5/1	3000	0.325	2-1/2
16/1	3300	0.25	3
16/1	3500	0.25	6
35/1	3500	0.17	1-1/4
45/1	3800	Did not extrude	
		Vacuum-Arc-Cast Billets	
7.4/1	3000	0.368	5-1/4
8.3/1	3500	1/8-3/4	4-3/4
16/1	3500	0.25	9
40/1	3800	0.158	10
45/1	3800	0.150	8

Thompson Ramo Wooldridge described[46] the extrusion of "3-inch diameter, high-carbon, low density" sintered tungsten billets at 3500 F with a reduction of 6/1. A banded structure was observed with grains nearer the surface being considerably more worked than those near the center.

Two attempts by Union Carbide to extrude sintered and unsintered slip-cast tungsten billets were unsuccessful. In both cases, discontinuous extrusions due to radial fracture occurred in attempted 4/1 reductions at 2300 F. A single attempt to consolidate 65-mesh tungsten powder in a 4/1, 2300 F extrusion was only partially successful. Higher reductions and temperatures were indicated[47].

Forging. The state of the art on the forging of tungsten and its alloys has recently been summarized by Thompson Ramo Wooldridge in their First Interim Report on the AMC Tungsten Forging Contract, AF 33(600)-41629[46]. As pointed out in this excellent survey, most of the large-scale development work on tungsten forging has been carried out using sintered billets and preforms aimed at the development of tungsten and tungsten-alloy rocket nozzles. In no instances of the Thompson Ramo Wooldridge survey or that conducted on this contract were experiences reported in which forged sintered billets or preforms were subsequently rolled to sheet or even converted to sheet bar. For this reason, the forging of sintered shapes is only briefly reviewed here. For a detailed review of recent progress in all aspects of tungsten forging, the reader is referred to the Thompson Ramo Wooldridge report.

The salient features of commercial forging experiences with sintered tungsten and tungsten alloy billets can be summarized as follows:

(1) Materials and Shapes. Aside from a few tungsten-molybdenum alloys of various compositions, all commercial forging experiences have been with unalloyed tungsten. Table 20 summarizes the degree of success reported.

Tungsten rocket nozzles and throat inserts ranging in sizes up to a 10-inch (84 plan inches) nozzle (weighing about 200 pounds) have been successfully forged. Also, the Allison Division of General Motors has successfully close-die forged plasma-arc-sprayed-and-sintered tungsten into rings having diameters of up to 12 inches and thicknesses of up to 1-3/4 inches. The workability of these rings at 2500 to 3000 F was described as "very good", and Allison has recognized the possibility of using this type of product as tungsten sheet bar[48].

Accumulated experiences indicate that a minimum sintered density of 90 per cent is necessary for reasonable yields, with densities of 92 to 95 per cent preferred.

Some difficulties have been observed in attempts to reproduce results obtained on smaller billets (less than about 3 inches in diameter) with larger billets. This "size effect" apparently varies with the billets from various producers and appears related to density variations from surface-to-center of the billets.

(2) Heating. Currently one of the most serious problems of the forging industry is the general lack of furnaces capable of heating large tungsten billets (diameters above 3 inches) and preforms to temperatures above 2600 F[46]. Induction heating appears to be adequate for heating at least small-diameter cylindrical billets. Two such units have been built and are being used successfully to preheat billets up to 3 inches in diameter to temperatures up to 4500 F without contamination[42,46].

TABLE 20. RESULTS OF COMMERCIAL FORGER TRIALS ON TUNGSTEN AND TUNGSTEN ALLOYS[46]

Forging Company	Composition, weight per cent	Consolidation Method	Success (Versus Cracking)	Comments
1	100W	Sintered powder	No	Lack of success attributed to maximum heating temperature of 2500 F
	60W-40Mo	Arc melted	No	
	50W-50Mo	Arc melted	No	
2	100W	Sintered powder	Yes	
	85W-15Mo	Sintered powder	No	
	70W-30Mo	Sintered powder	No	
	60W-40Mo	Sintered powder	No	
	50W-50Mo	Sintered powder	Yes	
	100W	Plasma arc cast	No	
	100W	Electron-beam melted	No	
	100W	Arc melted	No	
	30W-70Mo	Arc melted	Yes	Reports 30W maximum for successful direct forging
3	100W	Sintered powder	Yes	
	50W-50Mo	Sintered powder	Yes	More cracking tendencies than for 100W
4	100W	Sintered powder	No	Lack of success attributed to maximum heating temperature of 2600 F
	70W-30Mo	Sintered powder	No	
5	100W	Sintered powder	No	Lack of success attributed to maximum heating temperature of 2500 F

Most of the details of commercial forging practice on tungsten are proprietary. However, the Steel Improvement and Forging Company has reported[49] that its normal forging temperature for sintered tungsten billets is 2800 F. All billets are first preheated at 900 F. Reductions of the order of 50 per cent are attempted before reheating which is generally carried out in a gas-fired furnace. Working temperatures are reduced during forging and finishing is generally accomplished at 2450 F. Recrystallization annealing is not normally done although a final stress-relief anneal at 1950 F is used.

"Opinions regarding the effects of oxidation on the forgeability of tungsten vary widely, from 'metal loss is the only problem' to 'selective oxidation along grain boundaries precipitates cracking and the piece should be thoroughly protected throughout the operations'"[46]. Most forgers use some form of protection during heating although others accept metal losses through oxidation.

(3) Forging. Hammer forging has been used predominantly for tungsten due to its low specific heat and the high heating temperatures required. Initial working is conducted under compressive forces. Tensile deformation, such as that obtained in open-die side forging, is not generally attempted until the structure is adequately worked. "No pattern has as yet been established regarding proper die lubricants or protective coatings for the work piece."[46]

Steel Improvement Forging Company, Westinghouse, and General Electric have each reported the results of research on forging powder-metallurgy tungsten alloys.

At Steel Improvement, boron additions up to 2 per cent were found to definitely improve low-temperature forgeability although these same additions caused hot shortness "above 3000 F". Elemental boron was not so successful in improving properties as the addition of boron as borax[49].

The beneficial effect of small boron additions on improving the ductility of melted tungsten was apparently first noted by Just[50] as described earlier. Later patents by Fonda[51] and Laise[52] describe improvements in the nonsag and recrystallization characteristics of tungsten filaments through the use of boron nitride additions to tungsten powder.

Westinghouse[41] has successfully forged 3/4-inch-square bars of tungsten containing 4 and 5 per cent thoria additions to 50 per cent reductions in height on open dies at 3090 F. In this same work, open- and closed-die forging was attempted on alloys containing small amounts of ZrO_2, B_4C, and CbC additions with the results shown in Table 21. This work indicated that preheating temperatures were quite critical. All of the alloys broke up at forging temperatures below 3270 F and good results were only obtained above about 3360 F.

TABLE 21. RESULTS OF FORGING EXPERIMENTS ON SINTERED TUNGSTEN-ALLOY BARS[41]

			Forging Conditions			
			Open Flat Dies, Air Atmosphere		Closed Swaging Dies, Argon Atmosphere	
Composition, weight per cent	Bar Size[a]	Sintered Density, g/cm^3	Temperature, F	Results	Temperature, F	Results
$W\text{-}1ZrO_2$	A	17.8	3090	No good	--	--
$W\text{-}1ZrO_2$	B	17.1	--	--	3360-3690	Good
$W\text{-}0.2B_4C$	B	17.2	--	--	3360-3690	Good
$W\text{-}0.5B_4C$	A	17.4	3090	No good	--	--
W-0.1CbC	B	17.5	--	--	3360-3690	Good
W-0.4CbC	A	17.4	3090	No good	--	--

(a) A — 2 kg, 3/4 x 3/4 x 24 inches.
B — 1.27-inch diameter x 4.5 inches long.

Westinghouse[31] also reported the successful forging of sintered unalloyed tungsten and 50Mo-50W bars, originally 1-5/8 to 1-3/4-inch-diameter, to 3/4-inch diameter. Bar densities were 94 per cent of theoretical and both heating and forging were accomplished within an argon-filled chamber. Preheating temperatures ranged from 3310 to 3540 F and swaging dies were used to work the materials. The unalloyed tungsten worked with greater ease than the 50Mo-50W alloy. Other alloy billets of 50Mo-50W which contained a deliberate 0.01 per cent carbon addition could not be forged successfully under these same conditions.

An early report[53] by the General Electric Research Laboratory described the successful side forging of a small (initially 0.012 square inch), sintered 68.5W-30Mo-1.5Th alloy at approximately 3900 F.

Ingots

With two outstanding exceptions, attempts to directly forge or roll arc-cast tungsten ingots in diameters above about 2 inches have not been successful. On the other hand, the feasibility of producing tungsten sheet from ingot via extrusion and forging has been demonstrated.

The difficulty in working tungsten ingots appears primarily due to the large grain sizes obtained in conventional consumable-electrode and electron-beam melting practices.

Direct Rolling

The only successful efforts in direct rolling of arc-melted tungsten or tungsten alloys to a sheet product have been accomplished with very small single crystals and button-type ingots.

In work for the Special Weapons Center of the Air Force(14), Battelle was able to effect 50 per cent reductions in 1/8-inch-diameter single crystals by flat rolling at 1470 F. Edge cracking became pronounced at lower rolling temperatures (750 to 1110 F).

The remarkable effect of rhenium additions on improving the hot ductility of arc-cast button-sized ingots of tungsten was first demonstrated by Geach and Hughes(54). Later work at Battelle(55,56) has shown that, with rhenium additions of 21 to 30 per cent, 20 to 40 gram sized ingots could be directly rolled to 0.025-inch-thick strip at temperatures as low as 1830 F. These effects of rhenium have been ascribed(57) to a change in the oxide morphology, a reduction in oxygen solubility, and the promotion of mechanical twinning as a primary mode of deformation. In view of the high cost of rhenium (about $600 per pound) and its limited availability, these developments are almost academic so far as the production of large alloy sheet is concerned. Nevertheless, additional studies(58) indicate that similar effects in tungsten can be obtained through the use of less costly alloying additions.

Extrusion

Seven organizations have reported successes in the extrusion of arc-cast tungsten and tungsten-alloy ingots. The most extensive extrusion programs are those which have been conducted at the Wright Air Development Division Refractory Metal Working Facility and the National Aeronautics and Space Administration.

The most recent (October, 1960) data from WADD(42) are summarized in Table 22. These results are believed to be especially noteworthy since the techniques used are possibly the most advanced and this group has had the benefit of working with a greater number of arc-cast tungsten and tungsten alloy ingots than any other to date. Each of the billets listed in Table 22 was nominally 3 inches in diameter and 6 inches long, and most were extruded bare, i.e., without cladding, but with a glass lubricant. In all instances, alumina-coated extrusion dies (described earlier) were used. With the exception of unalloyed billets, 238 and 239 (which were melted by and returned to Universal Cyclops), all of the ingots listed in Table 22 were melted by and returned to the Climax Molybdenum Company.

These data show that tungsten and the tungsten-molybdenum alloys respond readily to the extrusion process. Of the 27 extrusions listed, only one stuck. The majority had good surfaces; 7 gave yields of 75 per cent or higher, and more than half gave yields of 50 per cent or better. These workers (personnel from the Harvey Aluminum Company) caution against generalizations from these few data and admit that the extrusion conditions were chosen cautiously because of anticipated difficulties.

Figure 16 illustrates the appearance of extruded Bar 238, which was extruded bare, and Bar 239 which was extruded in a mild steel liner. The roughened surface

TABLE 22. EXTRUSION DATA FOR ARC-MELTED TUNGSTEN AND TUNGSTEN-MOLYBDENUM ALLOYS(42)

WADD Facility, 3 x 6-Inch Billet Size

Serial No.	Composition, weight per cent W	Mo	C	Extrusion Ratio	Temperature, F	Pressure Start, tons	Yield, per cent	Comments
238	100	--	--	4/1	3000	441	85	Fair
239	100	--	--	4/1	3000	468	--	Smooth
254	100	--	--	4/1	3000	600	--	Fair
251	100	--	--	4/1	2800	590	82	Smooth
272	100	--	--	4/1	2800	540	63	Smooth
273	100	--	--	4/1	2800	542	55	Wrinkled
274	100	--	--	4/1	2800	600	70	Wrinkled
275	100	--	--	4/1	2600	548	--	Smooth
212	85	15	--	4/1	3000	695	79	Smooth
247	85	15	--	4/1	3000	630	71	Fair
248	85	15	--	4/1	3000	673	67	Smooth
269	85	15	--	4/1	3000	660	--	Rough
211	85	15	--	4/1	2900	--	--	Smooth
259	70	30	--	4/1	3000	645	--	Smooth
252	70	30	--	5/1	2900	613	22	Fair
276	70	30	--	4/1	2800	660	--	--
224	70	30	.006	4/1	2600	630	75	Smooth
225	70	30	.006	4/1	2400	665	55	Smooth
232	67	33	.01	4/1	3000	685	50	Smooth
230	67	33	.01	4/1	2600	650	78	Smooth
231	65	33	.02	4/1	2600	Stuck	0	Too stiff
208	50	50	--	4/1	2400	580	60	Smooth
277	50	50	--	6/1	3100	695	--	Smooth
222	50	50	.008	4/1	2200	592	69	Cracked, smooth
223	50	50	.008	4/1	2000	595	67	Smooth
227	50	50	.01	4/1	2200	598	75	Smooth
228	50	50	.01	4/1	2200	578	80	Smooth

Can removed Canned No can

a. Bar 239 b. Bar 238

FIGURE 16. EXTRUDED BARS OF UNALLOYED ARC-CAST TUNGSTEN

Courtesy of Universal-Cyclops Steel Corporation.

and "dog-bone" shape of the canned extrusion, after removal of the steel, appear to be characteristic of arc-cast tungsten after extrusion in mild or stainless steel cans[45]. By comparison, the surface of Bar 238 was quite smooth and a yield of 85 per cent was realized on this extrusion after conditioning. Metallographic examination showed approximately 15 per cent recrystallization had occurred during extrusion.

Figure 17 shows a plot of the extrusion constant, K*, versus temperature for tungsten, molybdenum, and a number of alloys extruded by WADD. The 85W-15Mo alloy line is shown dashed since this represents only one temperature of 3000 F. These data are of interest and suggest that tungsten-rich alloys containing 15 to 30 per cent molybdenum offer appreciably greater resistances to deformation (over the range of 2600 to 3000 F) than unalloyed tungsten.

While the work at NASA has been restricted to a lesser number of billets than that at WADD, this program has been more extensive in the range of extrusion variables covered. Thus, extrusion speeds have ranged from the medium range available with standard presses (up to 30 ips) to the high range obtainable on Dynapak units (up to 2500 ips). Extrusion temperatures ranged from 2300 to 4000 F, and reduction ratios varied from 5.5/1 with low velocity up to 45/1 at high velocity[45].

The low-velocity extrusion data are summarized in Table 23. Figure 18 illustrates extrusion billets typical of those used. Generally, columbium or stainless steel cans, plus glass coatings, were used as lubricants. As indicated in Table 23, with the use of these lubricants, reductions of 5.5/1 at 2300 F to 8/1 at 3100 F were effected although die washout was severe. Generally, surface roughness varied from fair, for the high-temperature columbium-clad extrusions, to poor for the stainless steel-clad extrusions. Metallographic examination showed that cold-worked structures were obtained in all extrusions, including the bars extruded at the highest temperature (3200 F), the maximum reduction ratio (8/1), and the most rapid extrusion speed (94 ipm).

The high-velocity extrusion data of NASA on arc-cast tungsten billets were presented in Table 19 and discussed earlier.

High-velocity extrusion of small arc-cast tungsten ingots and swaged rods has also been conducted successfully at the California Institute of Technology Jet Propulsion Laboratory[59]. The conditions used and results obtained are given in Table 24. These workers reported that each of the bars, extruded with reductions from 5/1 to 16/1 at 3500 F, were completely recrystallized.

A series of arc-cast tungsten-molybdenum alloys and one tungsten-tantalum alloy were also checked in a cooperative effort between NASA (who melted the ingots) and Thompson Ramo Wooldridge (who extruded them)[46]. Extrusion data for these billets are listed in Table 25, and extrusion constant data are given in Figure 19.

---

*$K = \dfrac{P}{A_0 \ln \dfrac{A_0}{A_1}}$,

where

K = resistance to deformation, psi

P = total force on the ram press, pounds

A_0 = cross-sectional area of container, square inches

A_1 = cross-sectional area of die opening, square inches.

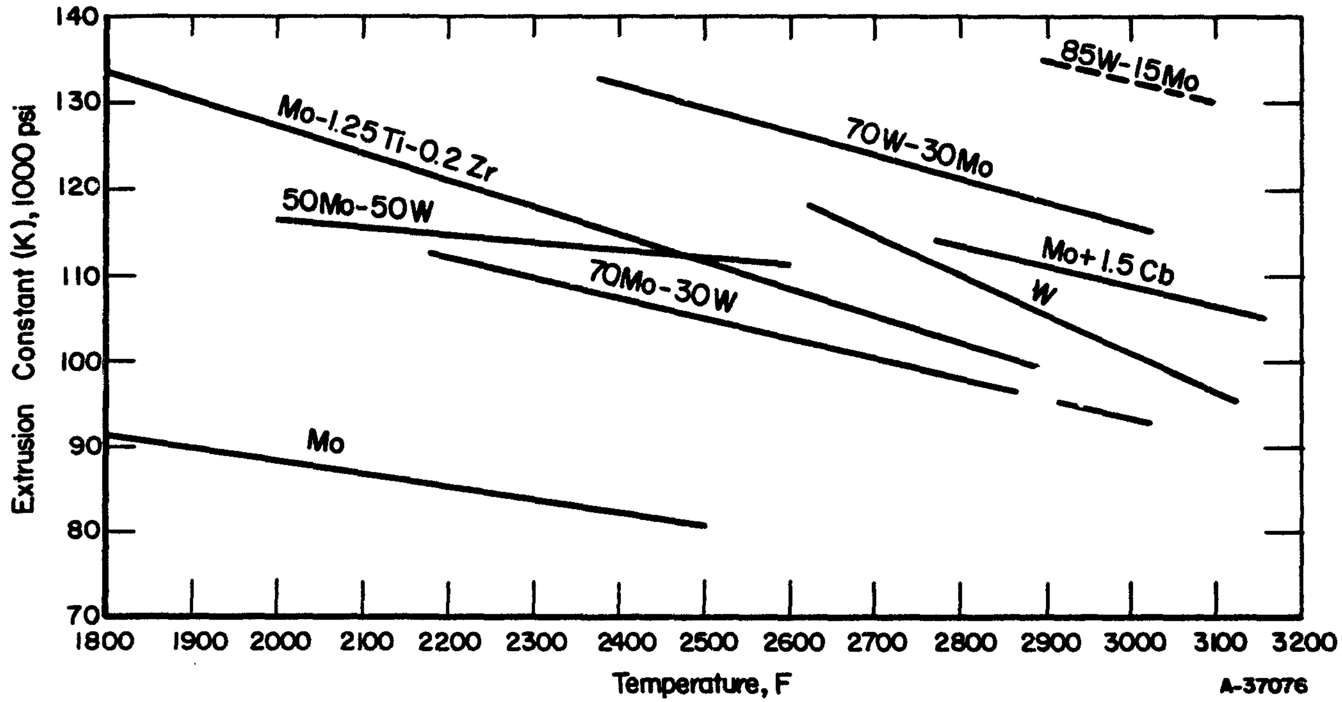


FIGURE 17. EXTRUSION CONSTANT VERSUS TEMPERATURE FOR TUNGSTEN, MOLYBDENUM, AND VARIOUS ALLOYS (ARC MELTED)(42)

TABLE 23. EXTRUSION DATA FOR UNALLOYED, ARC-MELTED TUNGSTEN(45)

Size of Unclad Billet, in. Diameter	Length	Cladding Material and Thickness	Reduction Ratio	Temperature, F	Extrusion Speed, ipm	Peak Value of K, psi
2-1/8	2-3/4	0.34-in. stainless steel	5.5/1	2300	70	74,900
2-1/8	2-3/4	0.34-in. stainless steel	5.5/1	2300	70	77,100
1-3/16	2-1/2	0.25-in. Cb	5.5/1[a]	3100	94	61,300
1-3/16	2-1/2	0.25-in. Cb	5.5/1[a]	3100	94	59,400
1-3-16	2-1/2	0.25-in. Cb	8/1	3100	94	53,000
1-1/2	3	None	5.5/1[a]	3200	Stalled	--
1-1/2	3	None	8/1	3600	Stalled	--
1-3/8	2-1/2	0.06-in. Cb	5.5/1[a]	3200	94	66,500
1-3/8	2-1/2	0.06-in. Cb	8/1	3200	94	60,800

(a) Reduction ratio for dies used on these billets was actually 6/1, but "washed out" to 5.5/1 for most of extrusions. K-value for these billets is based on initial breakthrough at 6/1 ratio.

FIGURE 18. UNALLOYED ARC-CAST TUNGSTEN EXTRUSION BILLETS

Courtesy National Aeronautics and Space Administration.

TABLE 24. HIGH-VELOCITY EXTRUSION DATA FOR UNALLOYED, ARC-MELTED OR SWAGED TUNGSTEN (59)

Jet Propulsion Laboratory 15/16 x 1-9/16-Inch Billet Size; 3500 F Preheat Temperature

Type of Tungsten	Reduction Ratio	Length of Extrusion in.	Fire Pressure, psi	Ram Weight, lb
Arc cast	5/1	4.7	500	1300
	5/1	5.8	500	2000
	10/1	11.0	550	1300
	10/1	12.2	550	1300
	10/1	11.0	600	1300
	16/1	5.6	400	1300
	16/1	16.0	600	1300
Swaged	5/1	5.5	500	1300
	10/1	9.5	550	1300
	16/1	13.0	550	1300

TABLE 25. EXTRUSION DATA FOR ARC-MELTED TUNGSTEN-MOLYBDENUM AND TUNGSTEN-TANTALUM ALLOYS(46)

Composition, weight per cent	Temperature, F	Reduction Ratio	Billet Pressure, 1000 psi	Extrusion Speed, ips
W-0.5Mo	3700	8/1	137.6	5
W-2.5Mo	3700	8/1	128.7	5/5.4
W-12Mo	4000	8/1	128.7	4.4/5.2
W-12Mo	4000	8/1	135.4	4/6.2
W-25Mo	3500	8/1	137.6	4.5
W-25Mo	3500	8/1	144.3	4
W-5Ta	4000	8/1	186.0	Stalled

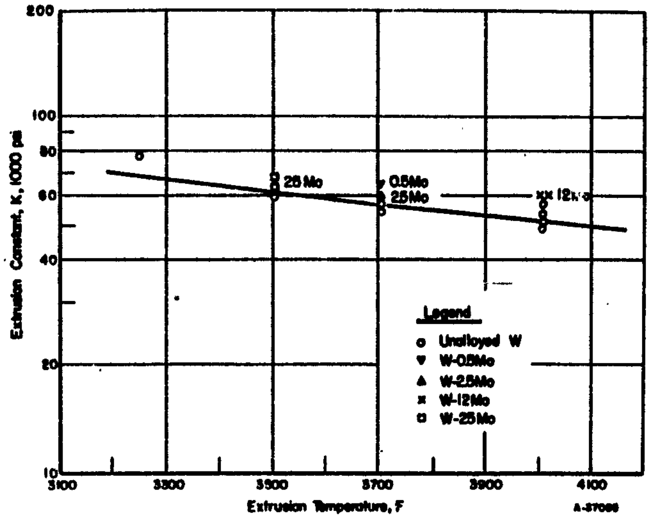


FIGURE 19. EXTRUSION CONSTANT VERSUS TEMPERATURE FOR TUNGSTEN AND VARIOUS ALLOYS (ARC MELTED)[46]

In their work for the Air Force, the Climax Molybdenum Company of Michigan has had considerable experience in the extrusion of alloys over the entire tungsten-molybdenum system. The results of their most recent studies[60] on tungsten-rich alloys are summarized in Table 26. All of these extrusions were conducted using glass lubricants and alumina-coated dies, and the yields and quality of the extruded bars reflect a considerable improvement over their earlier experience[33] using steel cans for a lubricant and high-speed steel extrusion dies.

TABLE 26. EXTRUSION DATA FOR ARC-MELTED TUNGSTEN AND TUNGSTEN-MOLYBDENUM ALLOYS

Climax Molybdenum Company, 3 x 6-Inch Billet Size[60]

Blank	Composition, weight per cent	Extrusion Temperature, F	Corning Glass No.	Reduction Ratio	Maximum Load, tons	Vickers Hardness Cast	Vickers Hardness Extruded	Recovery, per cent	Remarks[a]
3868-2	W-0.01C	3000	7052	4/1	590	357	464	81.6	1
3871-1	W-0.01C	2600	7052	4/1	548	333	NA	NA	--
3871-2	W-0.01C	2800	7052	4/1	540	333	446	62.9	1
3871-3	W-0.01C	2800	7740	4/1	542	333	464	55.1	2
H57-2	W-0.01C	2800	7740	4/1	600	351	459	70.4	2
3869-1	W-30Mo-0.01C	3000	7052	4/1	645	272	342	67.2	1
3869-2	W-30Mo-0.01C	2800	7740	4/1	660	272	--	0	3
3869-3	W-30Mo-0.01C	3000	7052	5/1	640	272	302	22.1	2,4,5
3790-5	W-50Mo-0.01C	3100	7740	6/1	695	232	--	0	3

(a) 1 - No surface machining required. Recovery reflects cropping and grinding losses only.
2 - Surface machining required. Recovery reflects machining losses as well as cropping and grinding losses.
3 - Sticker.
4 - Incomplete extrusion.
5 - Crack developed in billet on straightening prior to machining.

The General Electric Research Laboratory has also reported[12] the successful extrusion of two, high-purity (oxygen, nitrogen, and carbon contents of less than 10 ppm each) arc-cast tungsten ingots. These billets, of 3-1/2 inches initial diameter, were extruded at 3000 F with reductions of 3.18/1 and 5.5/1, respectively. Both extrusions were sound except for slight nose cracking and fish scaling near the butt ends.

Union Carbide has also reported success in extruding two 3-inch diameter unalloyed arc-cast ingots at 2300 F, using a double cladding of mild steel over stainless steel.[47]

The largest arc-cast tungsten ingot which has been successfully extruded was 6 inches in diameter. This ingot, prepared by Westinghouse, was machined to a 5-5/8-inch diameter billet and extruded at Canton Drop Forging and Manufacturing Company to a 3-inch-diameter bar at 2300 F. The billet was preheated in a salt pot and lubricated with a proprietary graphite coating. Reportedly, a steel dummy block was used and nose cracking was limited to about a 3/4-inch length. Portions of the extrusion have subsequently been forged and rolled at 2300 F "with good results".

More recently the first successful extrusion of tungsten sheet bar from arc-melted ingot has been reported[61]. This was accomplished by the Flight Propulsion Laboratory Department of General Electric on Air Force Contract No. AF 33(616)-7484. The billet used was machined from a 6-inch-long section of a 4-inch-diameter electron-beam-melted ingot, prepared by the Stauffer Chemical Company and contained their proprietary grain-refining addition. After machining this section to 3.35-inch-diameter, it was canned in molybdenum, heated for 2 hours at 3000 F, and extruded to a rectangular section of 0.62 by 2.87 inches. The resulting extrusion, "which appeared to be completely sound", was produced by a maximum force of 565 tons.

Forging

With two notable exceptions, success in direct forging arc-cast tungsten or tungsten-alloy ingots has been obtained only on small ingots or ingot sections. The exceptions include a 4-7/8-inch-diameter consumable-electrode arc-melted 60W-40Mo ingot and a 3-1/2-inch-diameter, electron-beam melted ingot containing a proprietary grain refiner.

The 4-7/8-inch-diameter ingot was prepared for the Martin Company by Oregon Metallurgical Corporation in a 6-7/8-inch length. After removal of an end crack, the ingot measured 4-7/8 inches high by 4-7/8 inches in diameter. As-cast grain size was established as ASTM 0-1 and hardness was found to be 196 BHN. The ingot was cogged, at 2960 F, by an outside vendor to a pancake measuring 9/16 by 11-1/4 inches, using four reheats. This billet has since been returned to Martin for attempted rolling to sheet starting at 3000 F.[62]

This success in working the arc-cast 60W-40Mo alloy is quite outstanding in view of the difficulties found by both Climax[33] and Westinghouse[31] in attempting to forge much smaller arc-cast ingots and samples of the 50Mo-50W alloy.

The 3-1/2-inch-diameter, electron-beam-melted ingot, made by the Stauffer Chemical Company, was closed-die forged by the Ladish Pacific Company to a 16-pound, rocket-exhaust entrance nozzle which had inside and outside diameters of 4 and 8 inches, respectively. Other forging tests on similar ingots made by Stauffer have been reported(46). In one case, a 3-inch-diameter ingot was reportedly upset from a 6 to 3-inch height in one blow.

All other experiences with the successful direct forging of arc-cast tungsten or tungsten-alloy ingots have been obtained on ingots or ingot sections of 2 inches or less. This is evident from a summation of the pertinent data from these works which is given in Table 27.

As indicated, most of the organizations attempting direct forging on small-sized samples of unalloyed arc-cast tungsten had moderately good success. Thus, reductions up to 67 per cent in height were obtained. On the other hand, attempts at direct forging even small arc-cast alloy ingots has been considerably less successful.

It is of interest to note that these combined experiences indicate that grain size may be the predominantly controlling factor in determining the workability of arc-cast tungsten. Thus, the only large (i.e., greater than 2-inch-diameter) ingots which have been successfully forged directly contain grain-refining additions (i.e., molybdenum, in the case of Martin's Oremet ingot, or the proprietary Stauffer addition in the electron-beam-melted ingots). Successes with small ingots or ingot sections could be expected on the basis of their inherently smaller grain sizes or more favorable grain orientation.

As shown in Table 27, experiences in converting arc-cast tungsten or tungsten-alloy ingot to sheet have been quite limited. Battelle's success in direct forging or rolling button ingots of W-(21-30)Re alloys to narrow strip has been described earlier. These results in tungsten are unique to the presence of large rhenium additions. On the other hand, experiences at Universal-Cyclops and the General Electric Research Laboratory have shown that tungsten sheet can be obtained from arc-cast ingot with the use of extrusion and/or forging as intermediate working operations to break down the cast structure.

At Universal-Cyclops, two 4-1/2-inch-long pieces were cut from the unalloyed extruded bar (238) shown in Figure 16 and were recrystallized by heating for 1 hour at 3000 F. One of these pieces was then forged at 2300 F to the 3/4 x 2-1/2 x 4-1/2-inch sheet bar shown in Figure 20. Minor grain-boundary bursts occurred on the end illustrated, but no other cracks were observed. After conditioning, the yield on the forging was 93 per cent. The sheet bar was then rolled at 2300 F to a defect-free 0.19 x 2-3/4 x 15-inch sheet as shown in Figure 21a. After another recrystallization anneal, rolling at 2300 F was again continued to produce the 0.040 x 6-1/2 x 17-inch sheet shown in Figure 21b. Some surface roughness occurred in this final rolling which was ascribed to the fact that the tungsten was sandwiched between two molybdenum sheets.

The second piece of this recrystallized extrusion was rolled directly to a 0.47 x 2-1/4 x 8-1/2-inch slab shown in Figure 22. This piece was sectioned into two equal sizes which were rolled to 0.090 x 6-1/2 x 6-1/2 inches. One of these was subsequently rolled to 0.020 x 13 x 13 inches after a recrystallization anneal. In these cases as above, rolling was carried out with a rolling temperature of 2300 F from a gas-fired furnace.

TABLE 27. SUMMARY OF RESEARCH PROGRAMS CONTRIBUTING TO FORGING OF ARC-MELTED TUNGSTEN AND TUNGSTEN ALLOYS

Organization	Alloy Composition, weight per cent	Ingot or Sample Size	Approximate Preheating Temperature, F	Forging Atmosphere	Subsequent Sheet Rolling Attempted	Subsequent Sheet Rolling Successful	Comments	Reference
Battelle	Unalloyed	1-1/4-in. diam x 9/16 in.	2800	Air	No	--	Upset 67 per cent in height	(15)
	W-(10-40)Re	30-g buttons	3000-3275	Air	Yes	Yes	21-30 per cent Re alloys forged and rolled to 0.030-in. strip	
Climax Molybdenum Co. of Michigan	W-(0-50)Mo	1-in. cubes	1800-2800	Air	No	--	Upset 50 per cent in height; study of cracking vs temperature	(33)
	W-0.05 Zr	1.7-in. diam x 1.5 in.	2600	Air	No	--	Cracked	(33)
Crucible Steel Co.	W-Ta-Mo-Cb	30 to 40-g buttons	>5000	Argon	No	--	20 different alloys upset 20 per cent in height	(63)
General Electric Research Lab	Unalloyed	1-11/16-in. diam	2700	Air	Yes	Yes	Initially extruded 3.18/1 at 3000 F	(12)
Linde	Unalloyed	3/4-in. diam	2200	Air	No	--	Single crystal; upset 75 per cent in height	--
Union Carbide	Unalloyed	1-2-in. diam x 1.5 in.	3100	Air	No	--	Upset 67 per cent in height; edge cracks only Side forged; cracks	(47)
Universal-Cyclops Steel Corp.	Unalloyed	1-1/2-in. diam x 4 in.	2300	Air	Yes	Yes	Initially extruded 4/1 at 3000 F	--

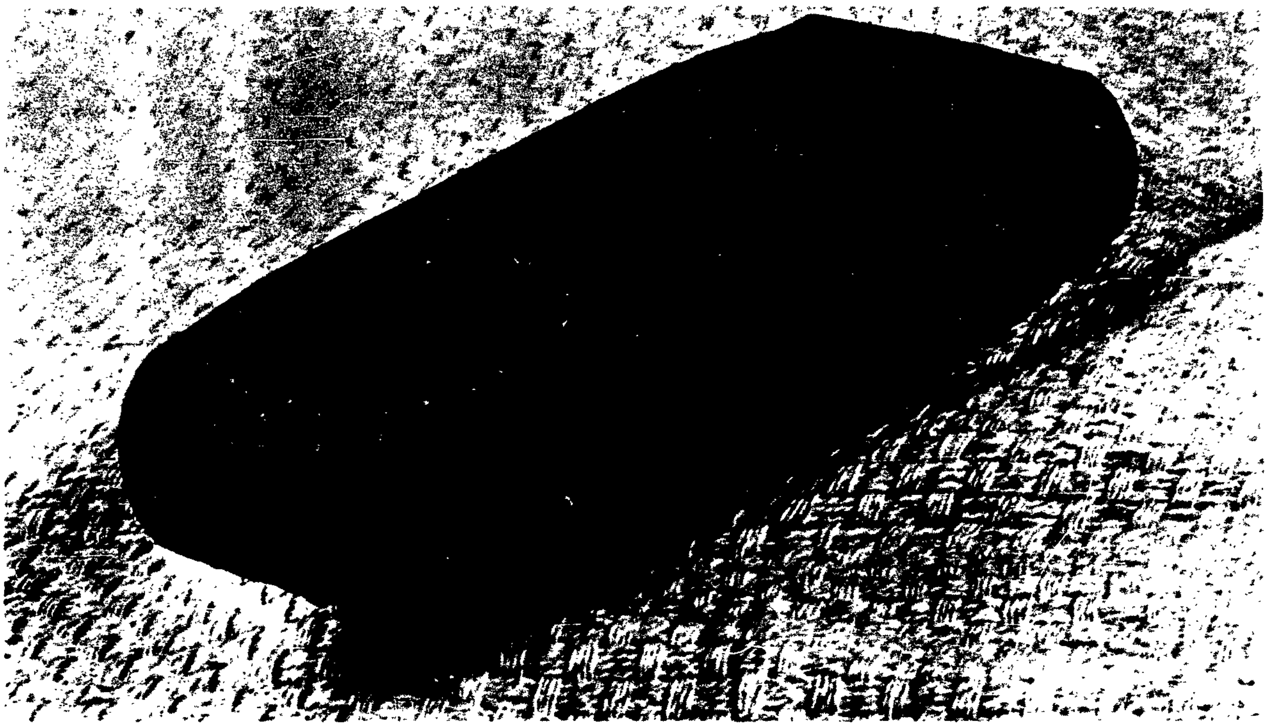
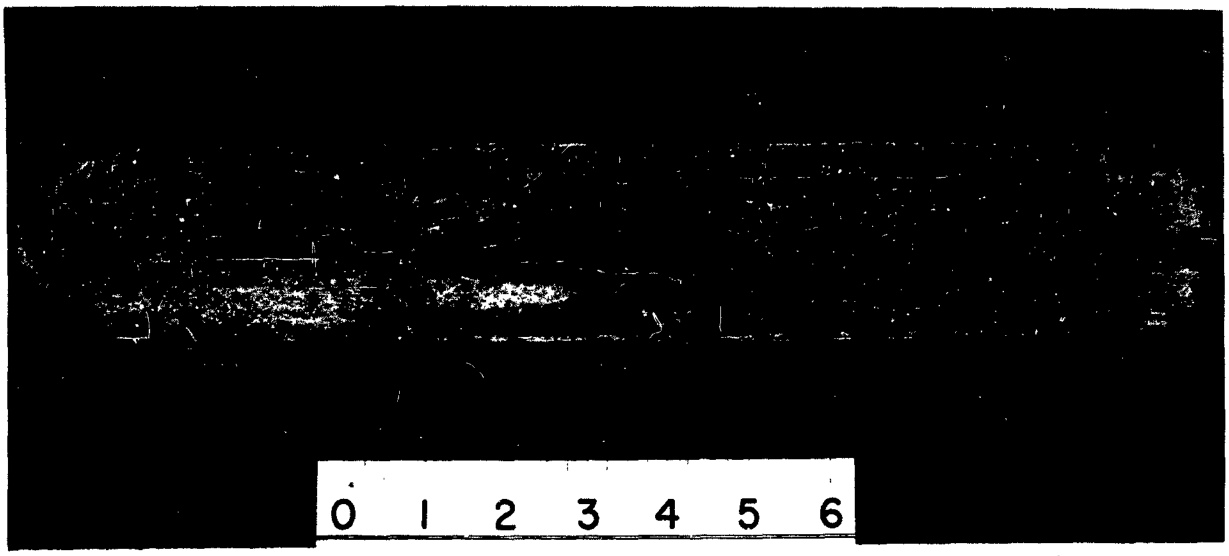
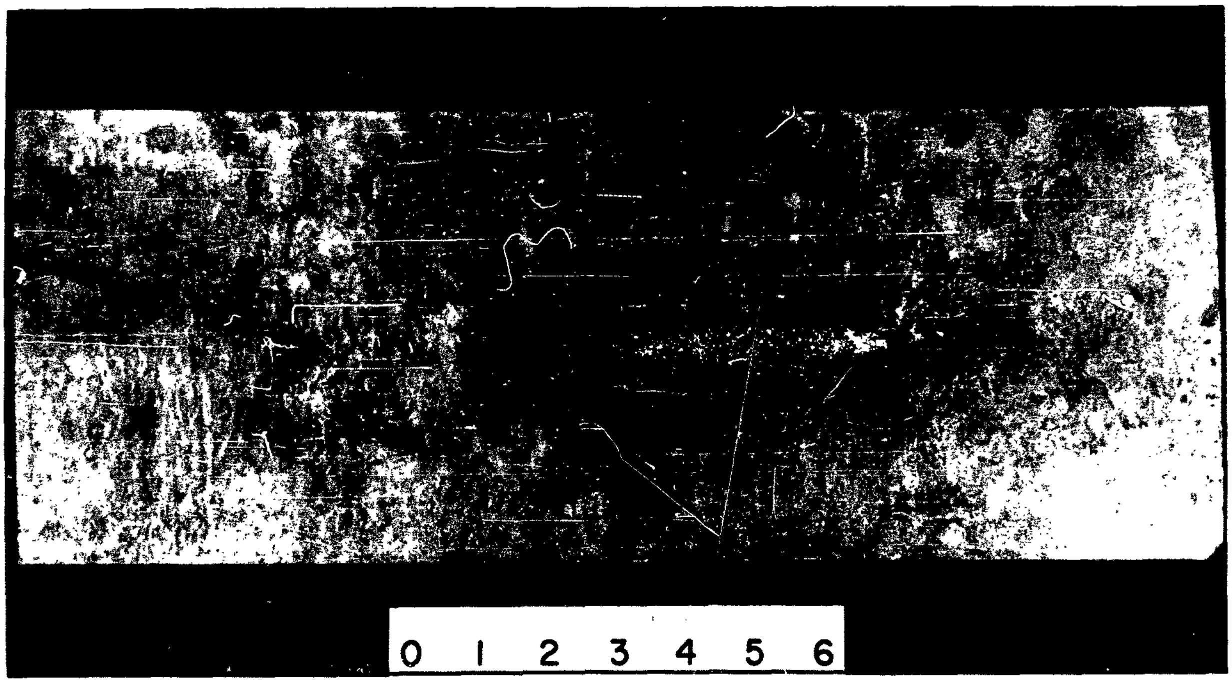


FIGURE 20. UNALLOYED TUNGSTEN SHEET BAR (3/4 x 2-1/2 x 4-1/2 INCHES), FORGED AT 2300 F FROM EXTRUDED, ARC-MELTED INGOT

Courtesy Universal-Cyclops Steel Corporation.



a. Sheet From Bar of Figure 20 After Conditioning and Rolling to 0.19 x 2-3/4 x 15 Inches



b. Sheet Shown Above After Recrystallization and Rolling to 0.040 x 6-1/2 x 17 Inches

FIGURE 21. UNALLOYED TUNGSTEN SHEET FROM ARC-MELTED INGOT, AS ROLLED AT 2300 F AFTER EXTRUSION AND FORGING TO SHEET BAR

Courtesy Universal-Cyclops Steel Corporation.

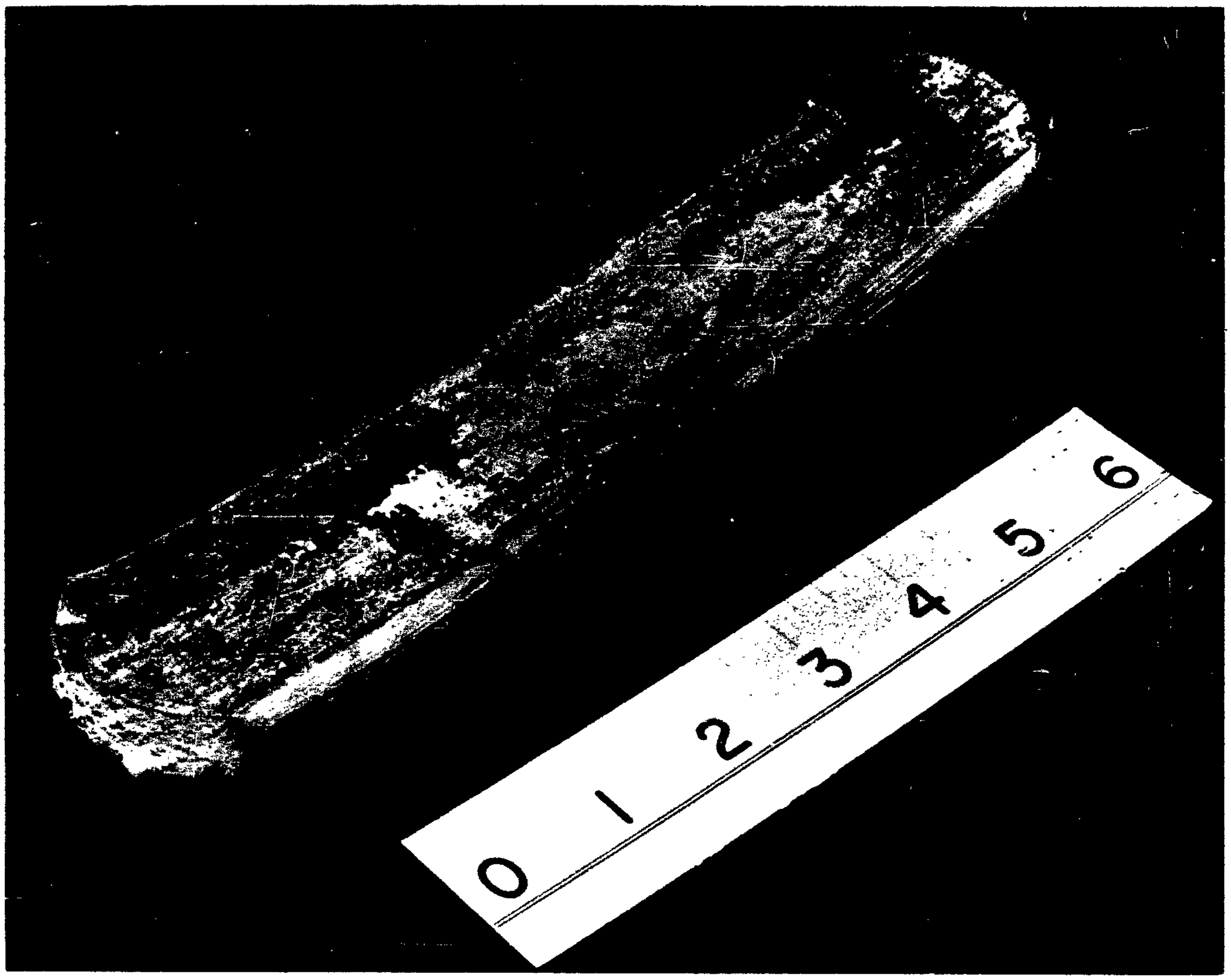


FIGURE 22. UNALLOYED TUNGSTEN SHEET BAR (0.47 x 2-1/4 x 8-1/2 INCHES) ROLLED DIRECTLY AT 2300 F FROM ARC-CAST INGOT AFTER EXTRUSION AND RECRYSTALLIZATION

Courtesy Universal-Cyclops Steel Corporation.

General Electric conducted several rolling experiments on portions of the two, high-purity, unalloyed, arc-melted tungsten extrusions described earlier.[12] In this work, slices were cut from the billet extruded at 5.5/1 and rolled directly at temperatures from 1650 F to 2370 F. At temperatures of 1830 and 2370 F, reductions up to 65 per cent were obtained without significant edge cracking. Extensive edge cracking did occur, however, at the lowest rolling temperature attempted (1650 F).

Castings

While the casting of tungsten and its alloys is not yet an established art, this method of consolidation shows future promise as a means of producing tungsten or tungsten-alloy sheet bar. This follows from a realization of the fine-grain sizes which have been obtained in the centrifugal ring castings of the 85W-15Mo alloy by the Oregon Metallurgical Corporation (see Figure 13) and from their success with rolling portions of these. Sections of these castings have been given a 50 per cent reduction by forging at 2730 F. After a 2-hour recrystallization anneal at 2910 F, rolling was initiated at 2550 F and continued with decreasing temperature. The results have been described as "very successful" and several small sheets of the 85W-15Mo alloy have been made in thicknesses down to 0.060 inch. Ultimately, Oremet hopes to perfect a process for slitting ring castings, then flattening them into a sheet bar for rolling.

To the present time, no successes in coverting slip-cast ingots or shapes into a wrought product have been reported.

POWDER METALLURGY VERSUS ARC CASTING

Relatively few organizations have had experience in making tungsten sheet from both sintered and arc-cast forms. Hence, it is difficult to draw effective comparisons in converting these materials. Nevertheless, the following comments can be offered on the basis of these materials as they are being produced today and the present conversion practices being used.

The inherently finer grain size of sintered tungsten sheet bar, when properly densified, lends this material the advantage of direct workability to sheet. The lack of furnaces for adequately sintering large sheet bars imposes a serious limitation on the size capability for this material.

Because of its initially finer grain size, the sintered product is generally regarded as having a greater tolerance for interstitial impurities. This stems from the associated facts that finer-grained material, which contains a higher level of interstitials than arc-cast material is directly workable while large-grain-sized arc-cast material generally is not. Hence, extrusion and forging appear to be essential in the preparation of sheet bar from arc-cast ingot.

After breakdown of the as-cast structure has been accomplished, the available evidence suggests that arc-cast material may be easier to fabricate than sintered. Thus, comparison experiences at Westinghouse and Universal Cyclops have indicated, respectively, that arc-cast material, when forged directly or extruded then forged, "moves more easily" and is not "as delicate" as sintered material, and arc-cast material appears to roll satisfactorily at appreciably lower temperatures. It is possible that these effects may be attributable to the purity differences in these materials.

It is of further interest to note that arc-cast ingot sizes are being made which can be used to produce sheet sizes at least equivalent to those now being made from sintered material.

PROPERTIES OF TUNGSTEN AND TUNGSTEN ALLOYS

Very few quantitative property measurements have been conducted on tungsten and tungsten-alloy sheet. Further, all such data have been obtained on material made by powder-metallurgical techniques. It has been shown that the composition, structure, and mechanical properties of the sintered product are quite sensitive to processing variables. This makes it difficult to directly compare the results of various investigators. Nevertheless, limited results with sintered and arc-melted tungsten (rod) suggest that arc melting may effect a significant improvement in the ductility of tungsten at temperatures above about 3000 F.

Physical and Thermal Properties

Selected physical and thermal properties for pure unalloyed tungsten are listed in Table 28. In comparison with other structural metals, tungsten is distinguished by its high melting point and density and low linear coefficient of thermal expansion.

Virtually no comparable physical-property data are available for any tungsten alloys.

TABLE 28. SELECTED PHYSICAL-PROPERTY DATA FOR UNALLOYED TUNGSTEN[64]

Melting Point, F	6170
Boiling Point, F	9900
Density, lb/in.^{3}	0.697
g/cm^3	19.3
Crystal Structure	Body-centered cubic
Lattice Parameter, A	3.158(3)
Specific Heat, cal/(g)(C)	
20 C (70 F)	0.033
1000 C (1830 F)	0.041
2000 C (3630 F)	0.047
Thermal Conductivity, cal/(sec)(cm)(C)	
20 C (70 F)	0.31
1000 C (1830 F)	0.27
2000 C (2910 F)	0.25
Linear Coefficient of Expansion, 10^{-6}	
20 C (70 F)	4.43
1000 C (1830 F)	5.17
2000 C (2910 F)	7.24

Softening and Recrystallization Behavior

Experimental work at Battelle[14] has indicated that interstitial contaminants, over the range normally encountered in commercial tungsten sheet, have little or no significant effect on hardness. Thus, single crystals of various purities were found to have hardnesses in the 340 to 360 VHN range. These hardnesses were only slightly below (about 10 VHN) those of fully recrystallized tungsten strip prepared by conventional powder-metallurgy techniques (Table 29). No hardness correlation to interstitial content* in the single crystals was possible. Hot-cold rolling increased these crystals' hardnesses to 450 to 500 VHN, the same range of hardnesses obtained on sintered tungsten samples prepared at Battelle and Westinghouse.[41] Upon full recrystallization annealing, the hardnesses of these samples returned to their original prewrought hardness level.

TABLE 29. EFFECTS OF INTERSTITIAL CONTENT AND PROCESSING HISTORY ON THE HARDNESS, RECRYSTALLIZATION, AND GRAIN-GROWTH BEHAVIOR OF UNALLOYED TUNGSTEN STRIP[14]

	Interstitial Content, ppm			Primary Recrystallization			Annealed 1 Hr at 3990 F	
Treatment	C	O	N	Temperature, F	Grain Size, grains/mm^2	Hardness, VHN	Grain Size, grains/mm^2	Hardness, VHN
				Electron-Beam Zone Purified[a]				
1-2 zone passes	20-30	<3	<0.5	2640-2730	20	350	5	335
5 zone passes	30-40	<0.2	<0.4	2370	50	375	10	335
7 zone passes	<35	20-40[b]	<0.9	2190	140	360	12	350
7 zone passes	100-200[c]	--	--	2370	200	400	20	348
				Sintered and Rolled[d]				
Vacuum sintered	49	55	<3	2910	650	366	650	367
H_2 sintered	55	49	<3	3090	1600	390	950	366

(a) Cold worked to 50 per cent reduction.
(b) Oxygen intentionally added.
(c) Carbon intentionally added.
(d) Cold worked to 75 per cent reduction.

The hardness values obtained for wrought and annealed samples of the K-100 tungsten sheet fall in this same range. Thus, as shown in Figure 23, the wrought hardness of this material decreased, with increasing annealing temperature, from about 498 to 380 VHN after complete recrystallization. As indicated in Figure 23, the onset of recrystallization in the K-100 tungsten sheet is accompanied by a rapid hardness decrease, but minimum hardnesses are not achieved until recrystallization is complete. Identical softening and recrystallization behavior were observed for a variety of sintered tungsten and tungsten-alloy strips in the Battelle work.

*Ranges, in ppm, covered were carbon (20-200), oxygen (<0.2-40), and nitrogen (<0.9).

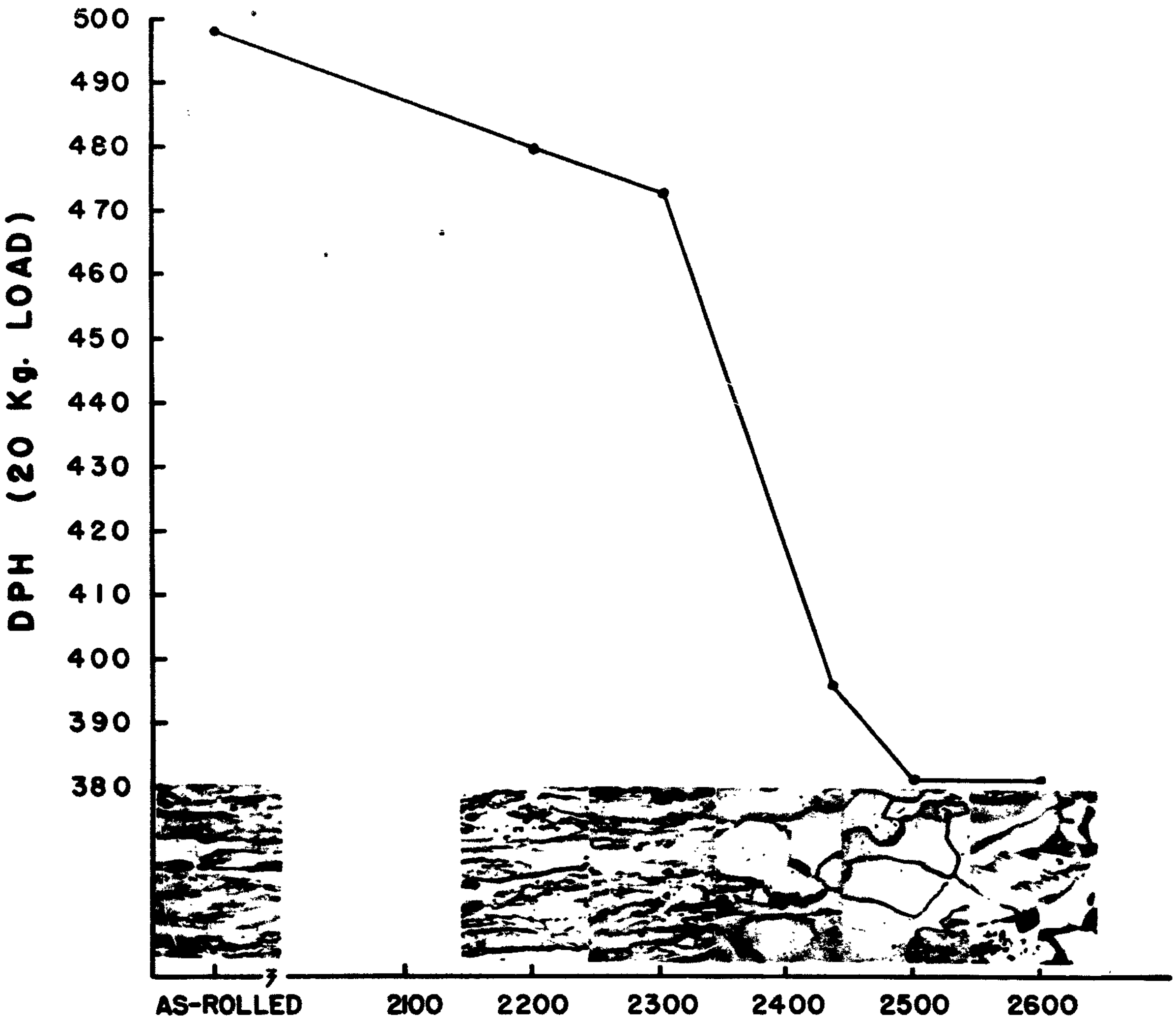


FIGURE 23. EFFECT OF 1-HOUR ANNEALING TREATMENTS ON THE HARDNESS AND STRUCTURE OF 65-MIL-THICK K-100 TUNGSTEN SHEET

Courtesy Sylvania Electric Products, Inc.

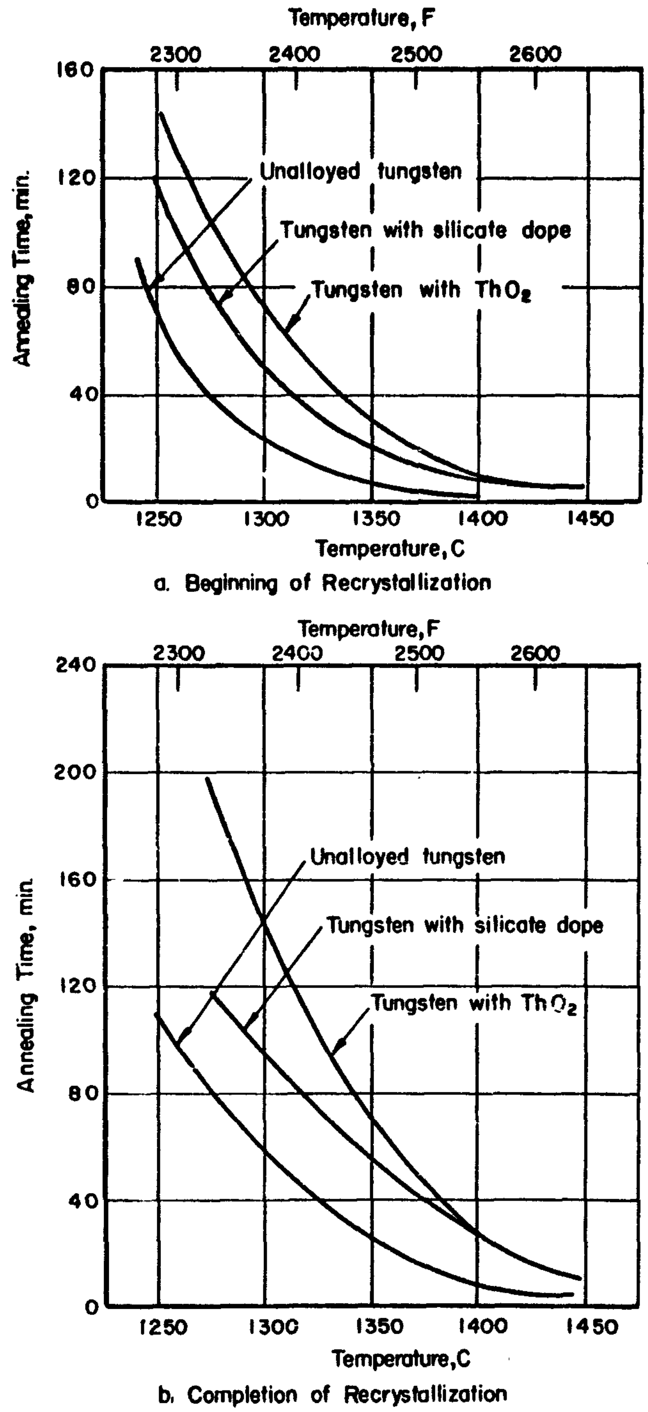


FIGURE 24. EFFECT OF SILICATE AND ThO_2 ADDITIONS AND TIME ON THE RECRYSTALLIZATION OF TUNGSTEN SHEET(65)

No definitive data are available on the effect of degree of cold working versus the recrystallization temperature of tungsten sheet. However, on the basis of experiences with tungsten rod and wire, one can fully expect the recrystallization temperature of tungsten sheet to decrease with increasing amounts of cold work.

As is the case with tungsten filaments, doping or thoria additions to tungsten sheet increase its recrystallization temperature. This is shown in Figure 24 by the data of Nachtigall[65]. These results were obtained on 30-mm (1.18-inch) square bars, sintered at 2730 F, and subsequently swaged, then flat rolled to narrow strip. The indicated 1 hour-recrystallization temperatures for the unalloyed, silicate-doped, and thoriated samples were 2370, 2445, and 2480 F, respectively. The 2475 F recrystallization temperature for Sylvania's K-100 alloy falls well in line with these values. This range of recrystallization temperatures, however, is about 500 F below the 2910 to 3090 F range obtained at Battelle on experimental strip (Table 29). While the reason for this divergence is not known, it is believed to result from the comparatively high sintering temperature (4710 F) and the small initial section size (1/4 x 1/2 inch) of the samples used in the Battelle work.

The data for Table 29 indicate that interstitial contaminants, in the ranges normal for sintered product, have no significant effect on either the recrystallization temperature or subsequent grain growth of tungsten. Conversely, work at both Battelle and Westinghouse[41] has indicated that trace metallic impurities exert a far more potent effect. Thus, the purification of tungsten by either successive zone-melting passes (Battelle data, Table 29) or by inert-electrode arc melting lowered the recrystallization temperature of both strip and rod samples by 700 to 900 F.

As illustrated in Figure 25, the grain-growth behavior of powder-metallurgy tungsten strip is related to both sintering atmosphere and annealing temperature upon subsequent vacuum heating from 3000 to 5000 F.

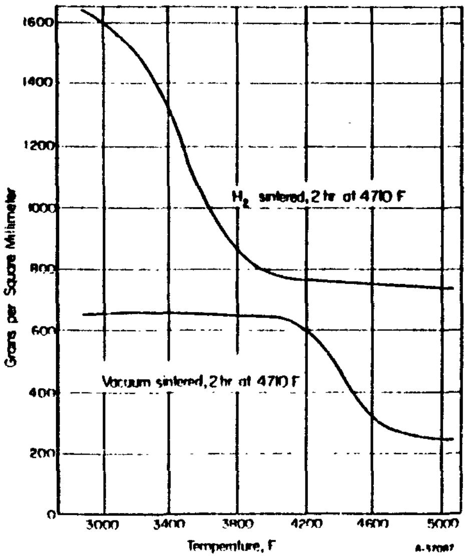


FIGURE 25. GRAIN SIZE OF WROUGHT (75 PER CENT COLD WORKED) POWDER-METALLURGY TUNGSTEN STRIP AS AFFECTED BY SINTERING ATMOSPHERE AND VACUUM ANNEALING[14]

The data of Table 30 show that the presence of finely dispersed inert oxides (e.g., aqueous ThO_2 plus Na_2O or 0.01-micron particles of ZrO_2) are effective in both increasing the recrystallization temperature of tungsten as well as increasing resistance to grain growth. Figure 26 shows representative microstructures obtained in this work.

TABLE 30. RECRYSTALLIZATION AND GRAIN-SIZE DATA FOR TUNGSTEN-DISPERSOID ALLOY STRIP[14]

Alloy Addition[a]	Approximate 1-Hr Recrystallization Temperature, F	Grain Size and Shape after 1 Hr at 3270 F	
		Grain Size, grains/mm^2	Grain Shape
Unalloyed	2910	600	Equiaxed
$1ThO_2$-$0.2Na_2O$(aqueous)	3270	1650	Equiaxed plus elongated
$1ThO_2$(aqueous)	2910	750	Equiaxed plus elongated
$1ThO_2$(3μ)	2910	1200	Equiaxed
$0.6ZrO_2$(0.01μ)	3270	900	Equiaxed plus elongated
$0.6ZrO_2$(1.9μ)	2910	850	Equiaxed
$0.25SiO_2$(0.02μ)[b]	2910	250	Equiaxed
$0.39MgO$(2.3μ)[c]	3090	45	Equiaxed
$1HfO_2$(1.3μ)[b]	2910	1150	Equiaxed

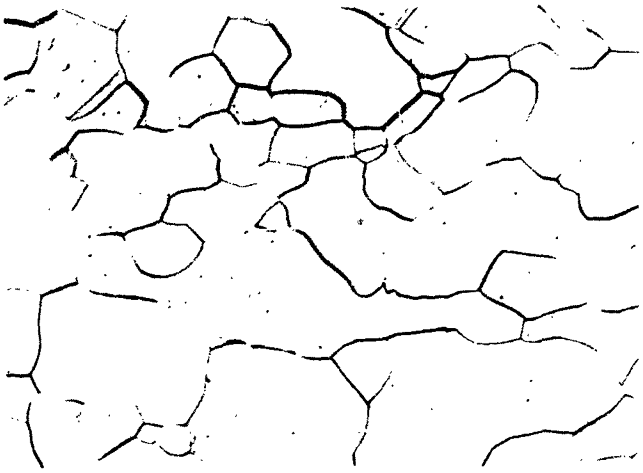
(a) All sintered 2 hr at 4710 F (unless otherwise specified) and hot-cold rolled to 75 per cent reduction.
(b) Sintered 2 hr at 5070 F.
(c) Sintered 2 hr at 5160 F.

Ductile-to-Brittle Transition

Like molybdenum and chromium, tungsten also shows a marked transition from ductile-to-brittle behavior with decreasing temperature. As with these other metals, the temperature at which this transition occurs is dependent on a number of factors including grain shape and size, strain rate, and metal purity. Thus, elongating the grain shape through cold deformation, decreasing grain size or strain rate, or improving metal purity all tend to lower the transition temperature.

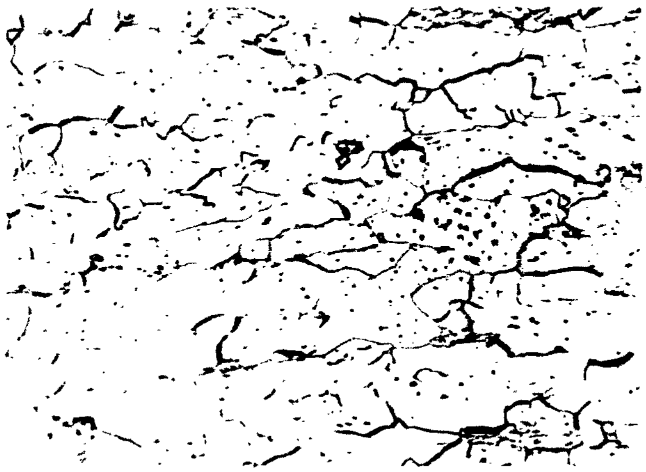
Room-temperature ductility can be achieved in small-diameter tungsten wires or narrow foil, chiefly by virtue of the extreme degree of cold working afforded through these somewhat unique fabrication conditions. However, no useful degree of bend or tensile ductility at room temperature has yet been achieved in more massive sections of the metal. Consequently, all plastic deformation on massive tungsten parts (e.g., the forming of tungsten sheet components) must be conducted at elevated temperatures to avoid brittle behavior.

Comparatively few quantitative data have been obtained on the factors known or suspected to influence the ductile-to-brittle transition temperature of tungsten sheet. On the other hand, several excellent studies have been conducted on the transition behavior of sintered- and swaged-tungsten rod.[41,66,67,68,69] Generally, these workers have shown that the transition-temperature zone for recrystallized tungsten



250X N60959

a. Unalloyed Tungsten



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b. Tungsten Containing (Nominally) 1 Per Cent ThO_2 Plus 0.2 Per Cent Na_2O

FIGURE 26. LONGITUDINAL SECTIONS OF TUNGSTEN STRIPS AFTER ANNEALING 1 HOUR AT 3270 F

Both materials were made by powder-metallurgical procedures and were initially hot-cold rolled to a 75 per cent reduction.(14)

rod tested at strain rates around 10^{-3}/sec lies toward the high end of a temperature range extending from about 350 to 850 F.[41,66,67] With decreasing strain rates, the transition temperature is lowered.[68]

For recrystallized tungsten rod, Atkinson, et al.,[41] offered the general correlation that improving metal purity decreased the tensile transition temperature. It was concluded that variations in the concentrations of oxygen (from 1 to 15 ppm), nitrogen (2 to 29 ppm), or hydrogen (from 1 to 3 ppm) were not the determining factors for ductility. Rather, variations in trace metallic impurities, especially nickel, were suggested.

The work of Pugh[66] and Ingram, et al.,[69] has shown that the tensile-transition temperature zone for wrought tungsten rod lies between 300 and 400 F which is below that obtained for recrystallized rod tested at the same strain rate.

The only transition-temperature data available on tungsten sheet have been obtained in bend tests. It is of interest to note, however, that these data indicate the transition temperature for ductile-to-brittle behavior in bending tungsten sheet is approximately the same as observed in tensile testing of tungsten rod. Thus, as illustrated by the curves in Figure 27, bend-transition zones of about 300 to 600 F are indicated for wrought strip and zones of about 550 to 850 F are indicated for recrystallized strip. Increasing grain size results in a further slight increase in the bend-transition temperature.

The results of 4T bend tests, using an included bend angle of 105 degrees, on wrought samples of Sylvania's K-100 doped tungsten sheet are summarized below:[40]

Sample	Bend Temperature, F	Remarks
1	480	Took full bend
2	390	Took full bend
3	390	Broke

These results compare favorably with the data of Figure 27 obtained on narrow, experimental strip of unalloyed tungsten.

The effects of alloying additions on the transition temperature of tungsten have been examined in studies at Battelle and General Electric. In one of the Battelle programs, Allen, et al.,[14] found that inert dispersed oxides can lower the bend-transition temperature of tungsten strip, primarily through grain-size control, in both the wrought and recrystallized conditions. The lowest bend-transition temperature observed (about 300 and 590 F, respectively, for the wrought and recrystallized conditions) were obtained with binary additions of 1 per cent ThO_2 and 0.6 per cent ZrO_2.

In other Battelle work[55,56], the effectiveness of rhenium in improving the low-temperature bend ductility of tungsten was demonstrated as summarized below:

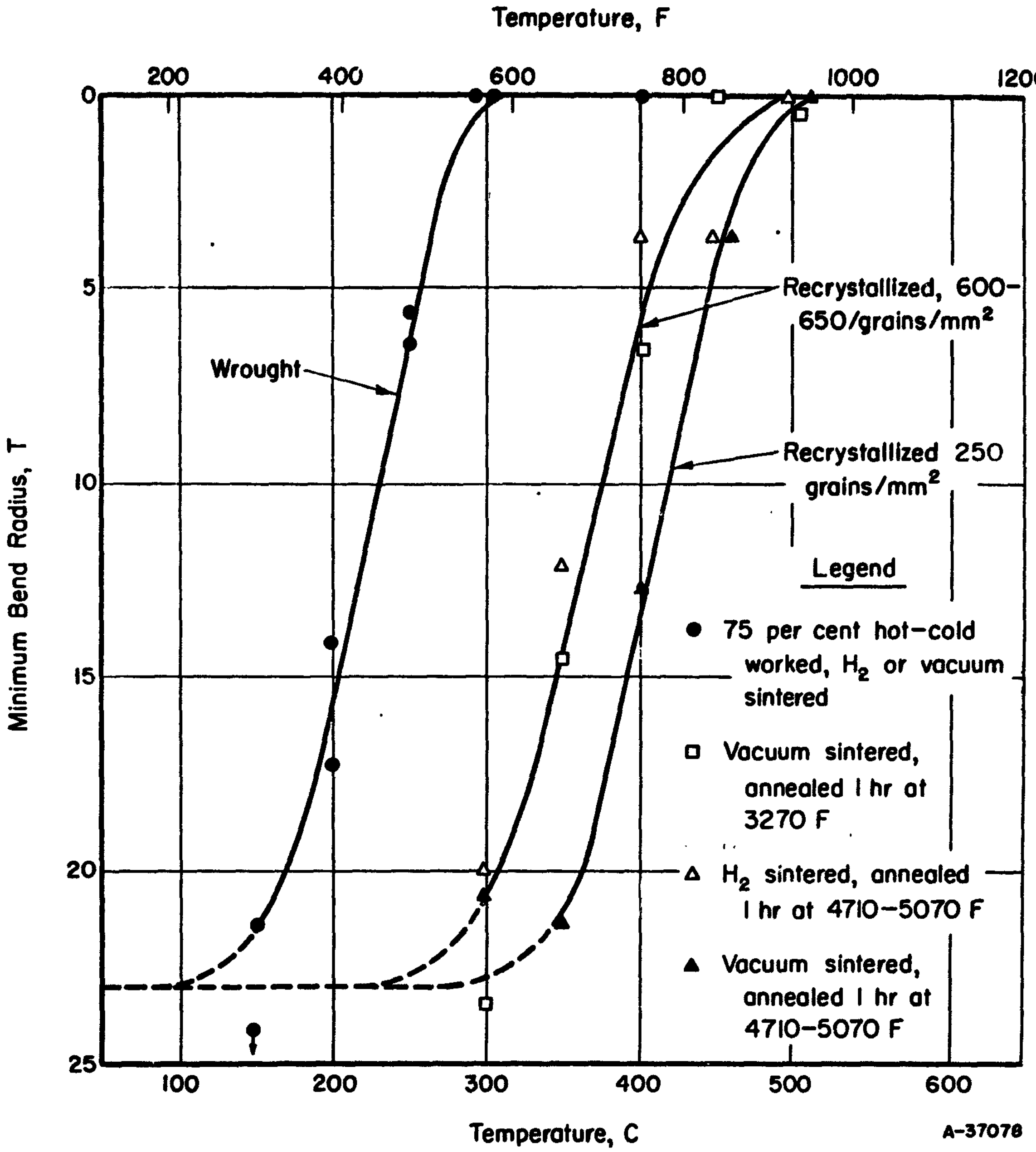

FIGURE 27. DUCTILE-BRITTLE BEND-TRANSITION TEMPERATURE OF POWDER-METALLURGY TUNGSTEN STRIP, SHOWING EFFECTS OF COLD WORK AND RECRYSTALLIZED GRAIN SIZE[14]

Rhenium Content, weight per cent	10T Transition Temperature[a], F	
	As Wrought	As Recrystallized
Unalloyed tungsten	375	625
22	--	480
24	--	255
26	--	50
28	--	50
30	-120	446

(a) The lowest temperature at which a sample can be successfully bent around a radius 10 times greater than the sample's thickness.

The above data for recrystallized samples show the optimum rhenium content lies around 26 to 28 per cent.

Investigations at the Flight Propulsion Laboratory Department of General Electric[70] were conducted using 1/4-inch-diameter bars prepared by powder-metallurgy techniques. The objective was to lower the tensile-transition temperature of tungsten by selective alloying with reactive metals to tie up the residual interstitial impurities as harmless stable compounds. The alloy compounds studied and results obtained are summarized in Table 31. It was noted that the transition temperatures of alloys sintered by radiation heating were generally lower than those obtained by direct resistance heating. As indicated in Table 31, a W-1.11Re alloy had the lowest transition temperature obtained. Also, tensile ductility could not be correlated to interstitial content.

TABLE 31. TENSILE-TRANSITION TEMPERATURES OF SWAGED, POWDER-METALLURGY TUNGSTEN-ALLOY RODS[70]

Alloy Addition, per cent	Interstitial Content, ppm				Sintering Condition[a]		Tensile-Transition Temperature[b], F
	C	O	N	H	Type Heating	Temperature, F	
None	10	6	<5	0.2	Radiation	2200-4200	265
0.10 Ti	15	640	<5	1	Ditto	2200-4200	275
0.014 Ti	10	96	<5	1	"	2200-4200	210
1.11 Re	10	13	<5	<1	"	2200-4200	160
2.98 Re	30	5	<5	<1	"	2200-4200	275
0.95 Ta	10	65	<5	<1	"	2200-4200	250
0.065 Zr	10	230	<5	1	"	2200-4200	275
None	10	22	<10	0.7	Resistance	4710	317
None	--	--	--	--	Ditto	4350	327
0.25 Y	11	21	<10	0.7	"	3990	418
0.07 Ti	9	34	<10	1	"	3990	351
0.21 Zr	10	66	<10	0.8	"	3990	312

(a) Vacuum atmosphere used.
(b) Designated arbitrarily as lowest temperature at which 5 per cent elongation was obtained.

Strength Properties

Strength data on tungsten sheet are extremely sparse. The main reason for this appears to lie with the difficulty of preparing sound tensile sheet samples due to the notch sensitive nature of the metal.

The only room-temperature tensile-ductility data reported were those of Sylvania for samples of the K-100 doped sheet of 0.065-inch initial thickness. A tensile elongation of 2.15 per cent was given for this material (apparently tested "as wrought") with a maximum stress of 182,900 psi.[40] The general effect of increasing the room-temperature strength of tungsten sheet through cold working has been shown by the Fansteel Metallurgical Corporation in the curve of Figure 28.

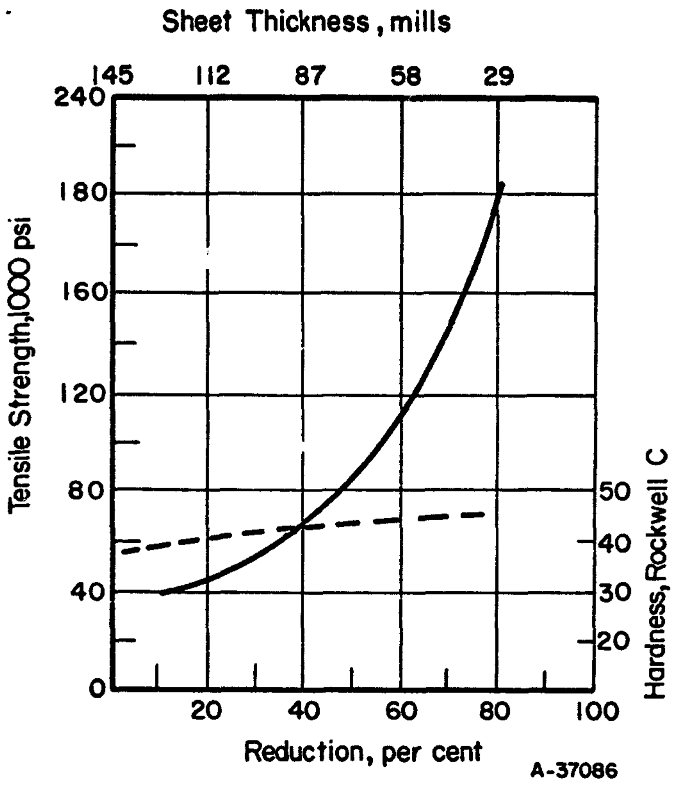

FIGURE 28. EFFECT OF COLD WORK ON THE TENSILE STRENGTH AND HARDNESS OF TUNGSTEN SHEET[64]

The tensile properties of unalloyed tungsten sheet were recently investigated by the Aerojet General Corporation[71] at temperatures from 4000 to 6120 F. In this work, the samples were self-resistance heated in argon and loaded incrementally by a timed flow of lead shot into a container provided for this purpose. Strain was measured by photographically recording the relative positions of tungsten fiducial wire markers spot welded within the gage length of the sample. All tests were conducted on wrought, 0.060-inch-thick tungsten sheet tensile samples provided by Fansteel. The data obtained are summarized in Table 32 and plotted in Figure 29.

TABLE 32. ELEVATED-TEMPERATURE TENSILE PROPERTIES OF SINTERED-AND-ROLLED UNALLOYED TUNGSTEN SHEET (0.060-INCH THICK)[71]

Test Temperature, F	Ultimate Strength, psi	0.2 Per Cent Offset Yield Strength, psi	Elongation, per cent	Loading Rate, psi/sec	Remarks[a]
4005	10,100	6,200	25	144	--
4401	6,800	4,100	14	128	--
4892	3,710	2,650	23	58	--
4937	4,510	3,200	16	133	--
5315	2,610	--	11	69	1
5373	2,880	1,530	14	128	2
5499	2,660	2,360	13	57	1
5679	1,970	1,650	7	40	1
5895	1,605	1,400	--	16	3
5931	1,705	1,160	2	47	2
5931	1,510	1,210	1	54	2
5976	1,405	1,100	--	42	2
6120 0	1,505	--	--	42	2

(a) 1 = Melting at fracture.
2 = Melting and oxide film at fracture.
3 = Oxide film at fracture.

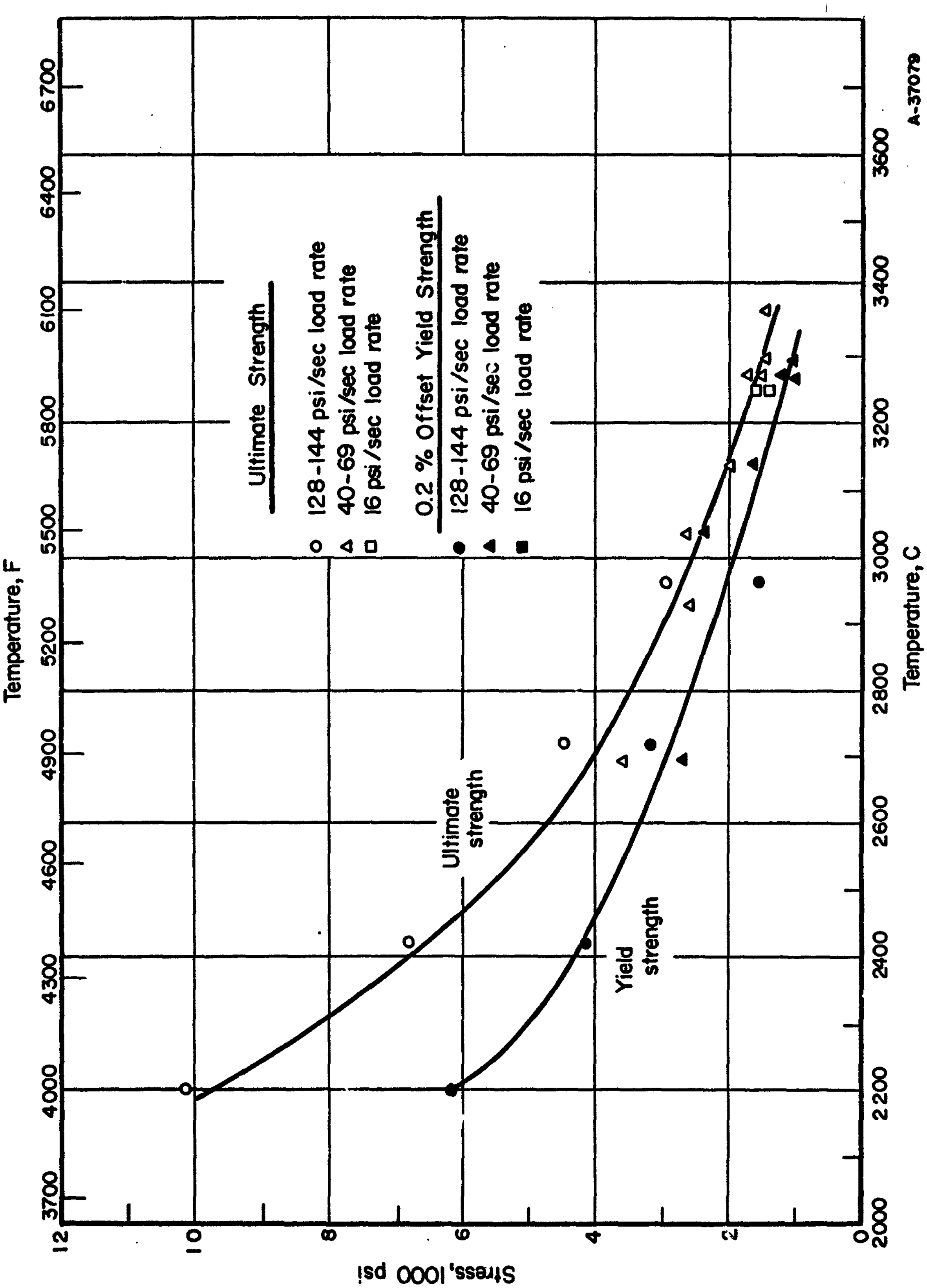

FIGURE 29. TENSILE STRENGTHS OF UNALLOYED TUNGSTEN SHEET (0.060-INCH THICK)(71)

For comparison purposes, the ultimate-strength data obtained from the sheet samples are also given in Figure 30, which contains a summary of data obtained at lower temperatures with tungsten and samples of widely variant history. At temperatures up through about 2500 F, the ultimate strength of tungsten appears quite sensitive to processing variables. At 2500 F, for example, strengths from 25,000 to 50,000 psi have been reported. With increasing temperatures, the effect of processing variables on ultimate strength appears to become less marked. Indeed, the curves of Figure 30 suggest that, above about 3500 F, the ultimate strength of unalloyed tungsten appears to be independent of both the consolidation practice used and prior thermal history.

On the other hand, the type of consolidation practice appears to have a marked effect on the degree of high-temperature tensile ductility obtained. This is reflected in the reduction-in-area values obtained on various rod samples as shown in Figure 31. The results of three separate studies(41,47,72) have shown that, at temperatures above about 2500 F, the tensile ductility of the powder-metallurgical product decreases drastically. Anomalous creep results with sintered product at 2500 to 2700 F have been attributed to this effect.(41) For arc-melted material, high reduction in areas are maintained to at least 4000 F. While the reason for this behavior is not known, these differences have been attributed to variations in impurity content between the powder-metallurgical and arc-cast products.

Most of the available elevated-temperature tensile data for wrought tungsten alloys are summarized in Figure 32. For the most part, these data represent the results of tests from single bars or heats, and a wide range of processing variables and test conditions are represented. Despite these qualifications, several general comments concerning the effects of alloying additions on the tensile strength of tungsten can be offered.

At temperatures up to about 3500 F, the strength of tungsten can be significantly improved by a variety of alloying additions. These include such solid-solution strengtheners as molybdenum and perhaps columbium and zirconium, as well as dispersoid strengtheners typified by ThO_2 and TaC. In comparison, an alkali silicate doping addition showed no strength advantages over unalloyed tungsten at 2500 F.(41)

Molybdenum is the only solid-solution strengthener which has been investigated in substantial amounts in tungsten. Alloys containing 10 to 25 per cent molybdenum have very similar strengths to about 4000 F and are somewhat stronger than tungsten from about 2600 to 3400 F. At higher temperatures, these alloys apparently have less strength than the unalloyed metal.(73) The 50Mo-50W alloy appears to offer no high-temperature strength advantage over pure tungsten.

A few results obtained by Union Carbide(47) indicate that small additions of columbium or zirconium give significant strengthening to tungsten at 3000 F. However, as noted by Hall(73), the rapid drop in strength of the W-0.88Cb alloy with increasing temperatures to 3500 F indicates that more complex alloys may be required to retain high strength in tungsten above these temperatures.

The most consistent improvements in the hot strength of tungsten above 2500 F have been obtained with the ThO_2 and TaC dispersoid additions.

The results of work at NASA(73) indicate that the addition of 1 per cent ThO_2 to tungsten raises its strength considerably. For example, unalloyed tungsten has a

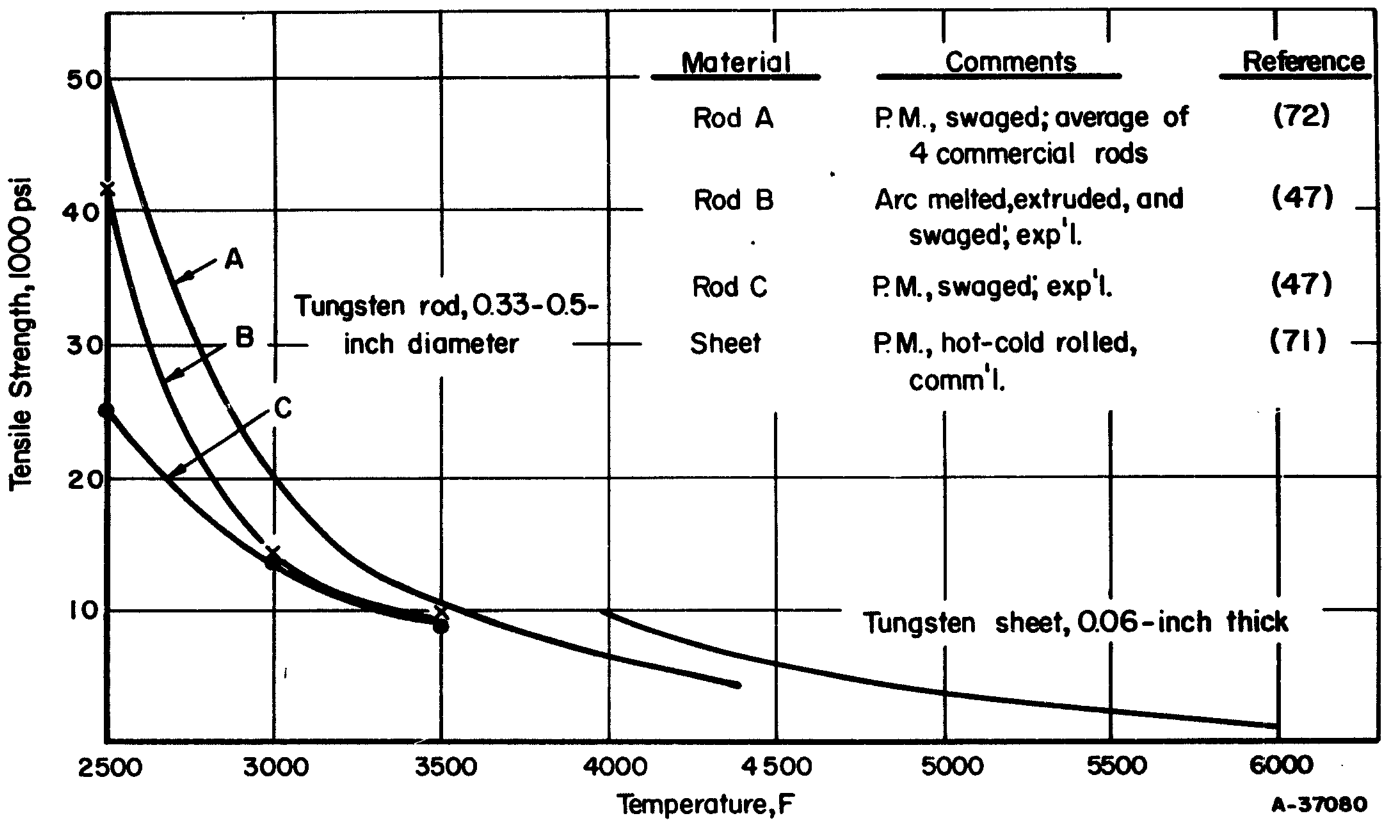

FIGURE 30. EFFECT OF TEMPERATURE ON THE TENSILE STRENGTH OF WROUGHT, UNALLOYED TUNGSTEN ROD AND SHEET

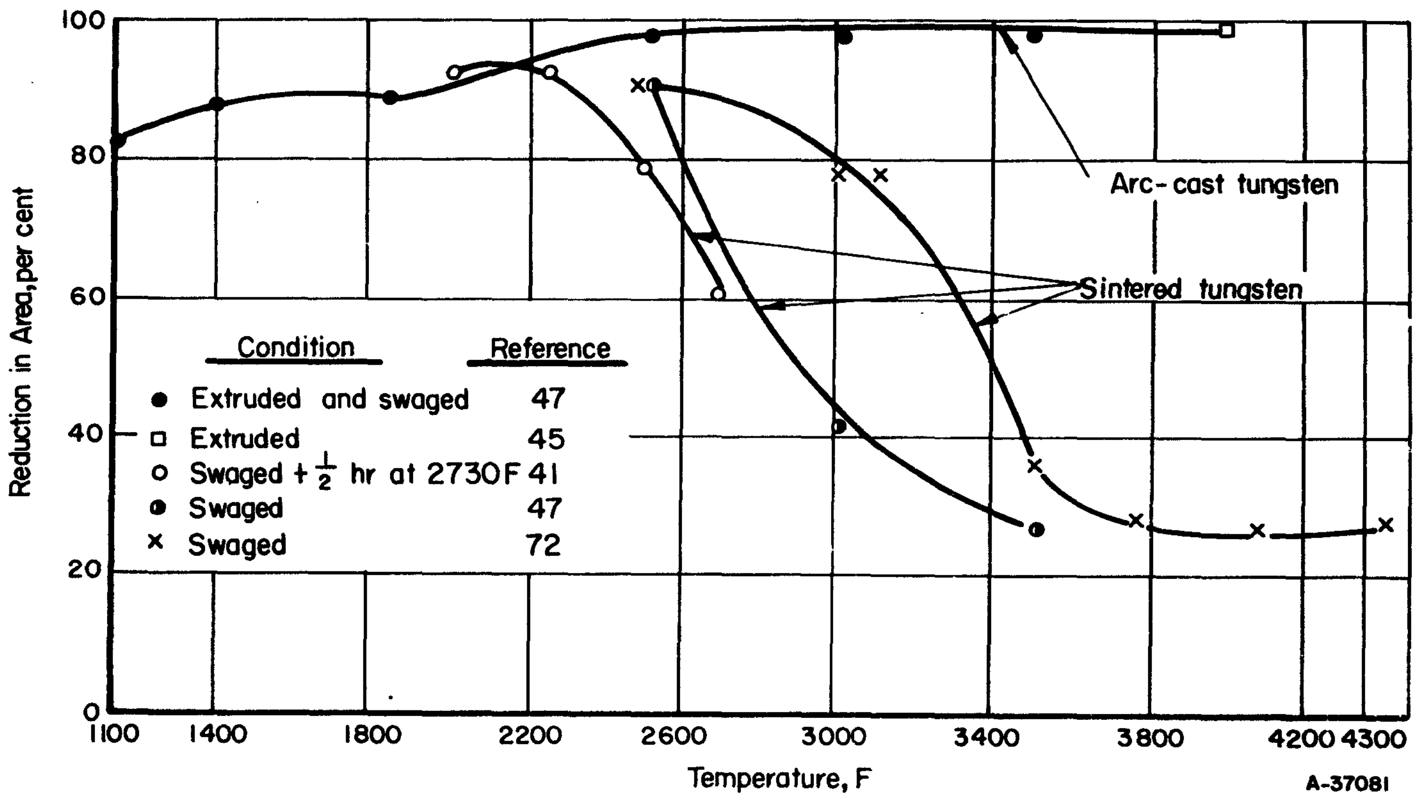

FIGURE 31. EFFECTS OF TEMPERATURE ON THE DUCTILITY OF ARC-CAST AND POWDER-METALLURGY TUNGSTEN(46)

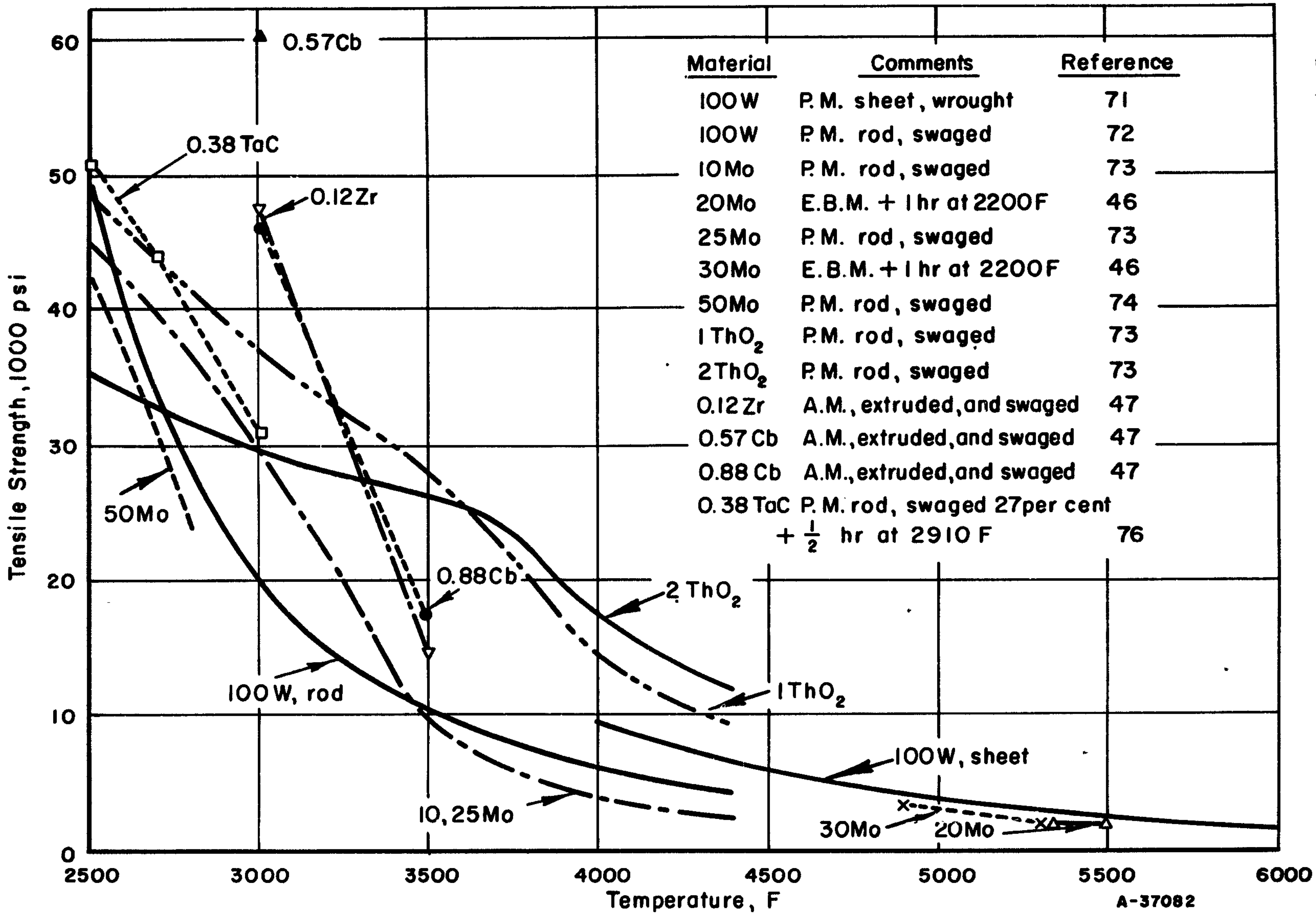

FIGURE 32. EFFECT OF TEMPERATURE ON THE TENSILE STRENGTH OF TUNGSTEN ALLOYS

tensile strength of 10,000 psi at 3500 F; the 1 per cent ThO_2 alloy falls to this level only above 4200 F; increasing the ThO_2 content to 2 per cent gives a further strengthening advantage as illustrated in Figure 32.

The effects of thoria additions at levels of 2, 4, and 5 per cent have also been studied at Westinghouse.[41,75] This work has generally shown that, at 2500 F, superior tensile and creep strengths were obtained in the 2 per cent ThO_2 alloy. This was apparently the result of a better dispersion of thoria at the lower level[41] and suggests that no further advantages may be gained by increasing the amount of thoria beyond 2 per cent.

At temperatures from 2500 to 3000 F, Westinghouse has found the W-0.38TaC alloy to have consistently higher tensile strengths than the W-2ThO_2 alloy, although this strength superiority diminishes rapidly as the test temperature approaches 3000 F.[75] At temperatures above 3500 F, the only alloys which appear to have a significant strength advantage over unalloyed tungsten are the 1 and 2 per cent thoria alloys.

The stress-rupture properties of unalloyed tungsten bar stock have been determined at 1600 to 2000 F by Pugh[66], 2200 to 2700 F by Atkinson, et al.[41], and from 4080 to 5070 F by Green[76]. Most of these data have been summarized in Figure 33. As noted earlier, the results of tests by Atkinson, et al., at 2500 and 2700 F showed considerable scatter and no correlation of the data could be obtained. A large gap in the information available on stress-rupture properties exists between 2200 and 4000 F.

The results obtained by Atkinson, et al., on creep testing thoriated alloys at 2500 and 2700 F are summarized in Figure 34. Generally, superior properties were obtained in the W-2ThO_2 alloy which had indicated stresses for a 100-hour life at 2500 and 2700 F of about 22,000 and 11,000 psi, respectively. None of the thoriated alloys showed evidences of the anomalous "hot shortness" observed in samples of unalloyed tungsten creep tested under these conditions.

Oxidation Behavior

The system tungsten-oxide is not well established because of its complexity. The principal oxides formed are $WO_{2.00}$, $WO_{2.75}$, $WO_{2.90}$, and $WO_{3.00}$, and each shows only a limited range of solid solubility for oxygen.[77]

When tungsten is heated in air, a lower oxide tarnish begins to form at temperatures below about 1300 F. Above this threshold-temperature region, the firmly adherent blue-black lower oxide begins to give way to the loose, yellow nonprotective oxide WO_3. As long as an underlying protective lower oxide remains, rate curves are parabolic in nature, but within a short time at temperatures above about 1300 F complete conversion to yellow WO_3 occurs and oxidation rates become linear. At temperatures above about 2200 to 2375 F, WO_3 is lost by evaporation as fast as it is formed, and the oxidation begins to parallel the combustion of graphite.

The oxidation rate of tungsten is particularly sensitive to increases in oxygen or water-vapor partial pressures. However, tungsten is relatively insensitive to attack by

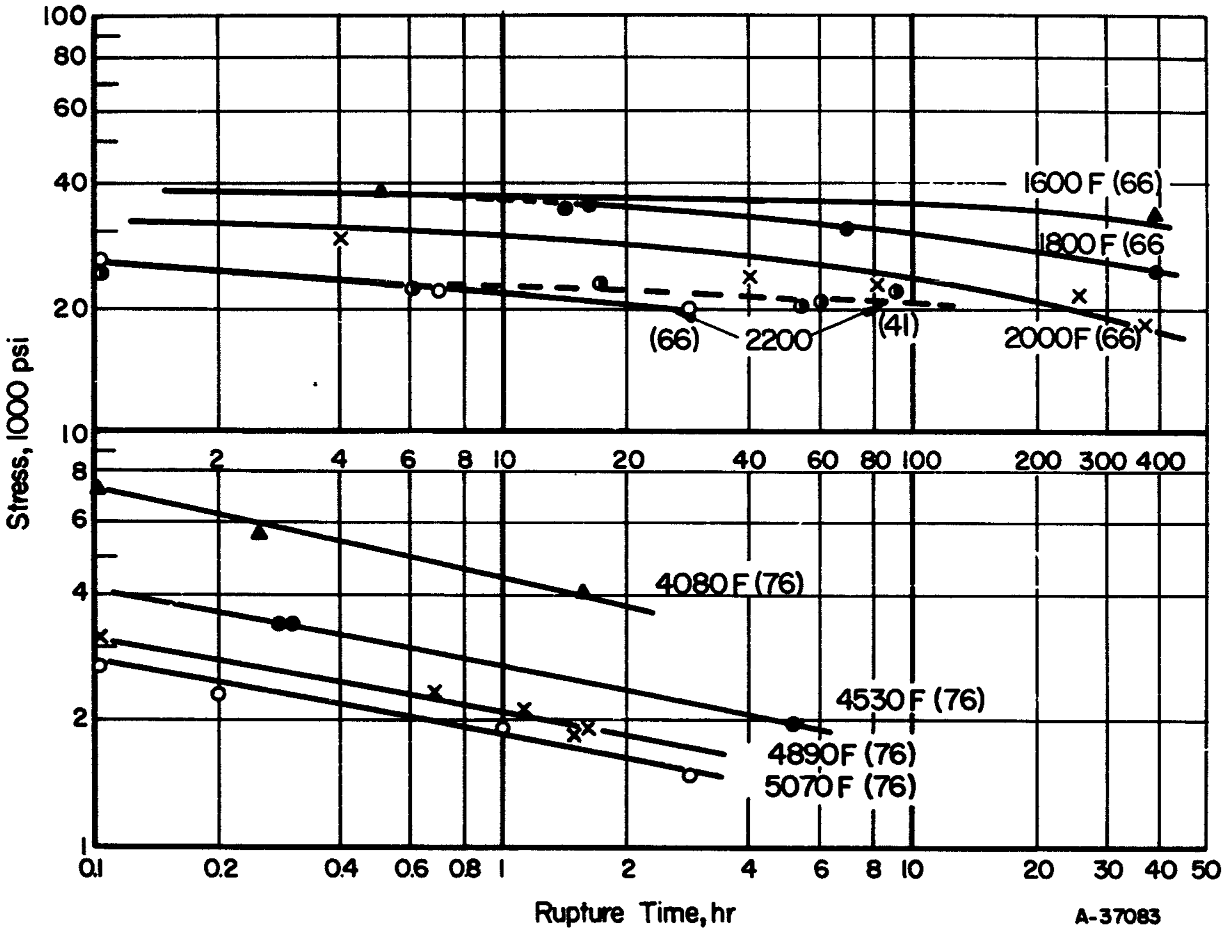

FIGURE 33. STRESS-RUPTURE PROPERTIES OF UNALLOYED TUNGSTEN ROD

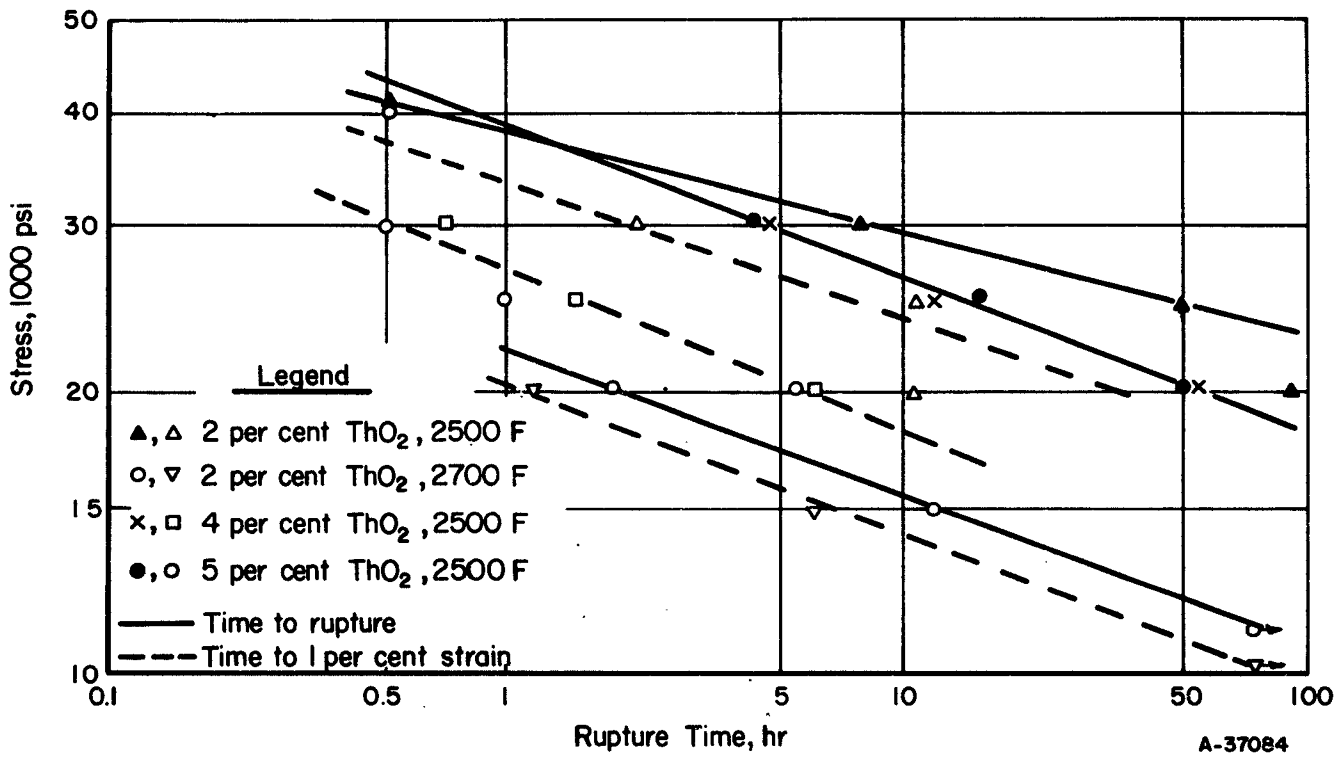

FIGURE 34. CREEP-RUPTURE PROPERTIES OF THORIATED TUNGSTEN BAR ALLOYS(41)

nitrogen. In general, tungsten is not so rapidly oxidized at high temperatures as is molybdenum, but in turn, is attacked at somewhat greater rates than are columbium and tantalum. From a fabrication standpoint, the oxidation of tungsten is apparently confined to the metal surface so that embrittlement during working at incandescent temperatures in air is not generally regarded as a serious problem.

Only a few oxidation tests have been conducted on any of the current, structural-alloy candidates. Results obtained by Climax[33] on binary tungsten-molybdenum indicate that molybdenum additions over the range of 2 to 40 per cent actually increase the oxidation rate of tungsten at 1750 F. Also, limited tests at Westinghouse[41] show that thoria additions up to 4 per cent gave no significant improvement in the oxidation behavior of pure tungsten at 2500 F.

It does not appear likely any of the other dilute, wrought tungsten-base alloys will show significantly improved oxidation resistance over unalloyed tungsten. For this reason, protective coatings will almost certainly be required for tungsten and its alloys to serve adequately in oxidizing environments at high temperatures.

TESTING AND INSPECTION PROCEDURES

Nondestructive Testing

Although there are some variations in details of testing and inspection procedures from one facility to another a marked over-all similarity exists. The nondestructive inspection procedures in common use for tungsten ingots or sintered tungsten rounds include density measurements, dye-penetrant checks (using for example, Turco Red dye), contact and/or immersion ultrasonic inspection, and Zyglo inspection techniques.

For sintered billets, immersion density measurements are not ordinarily made on shapes of less than 90 per cent of theoretical density. One organization restricts dye-penetrant checking on sintered shapes to those of greater than 80 per cent density. Of the above tests, each organization normally carries only those which are appropriate to the particular operation involved, or for which equipment is available. For example, the use of ultrasonic and Zyglo inspection procedures is not universal.

In the present state of the art, inspection procedures are not as well established as those used, for example, in connection with steel production. Further, some discretion may be called for in the acceptance of results. For example, density measurements on sintered rounds yield average densities, and give no clue as to homogeneity of density.

The present state of interstitial impurity analysis in tungsten has been summarized by Mallett[78]. It is of further interest that the Materials Advisory Board Refractory Metals Sheet Rolling Program Subpanel on Analytical Techniques is conducting a survey of analytical techniques for use on tungsten as well as on molybdenum, columbium, and tantalum. An effort is being made to assess current problems of analysis and to develop solutions for them.

In the determination of carbon in tungsten, the conductometric method is by far the most common procedure used. Of 17 facilities listing analytical procedures, 16 reported the use of the conductometric method. One producer reported the use of a titration method. Practically all those reporting conductometric methods used Leco equipment. Two organizations also used, in addition, volumetric methods for carbon determination.

According to questionnaire responses, some type of vacuum fusion is commonly used to analyze for oxygen. Both inert-gas and vacuum-fusion methods are used with about twice as many vacuum as inert-gas procedures in existence. Conductometric determination is largely used to measure CO_2 produced. Volumetric and isotopic dilution were also reported in use in two separate instances. Leco apparatus is in common use for oxygen analyses also. One organization uses fractography on coarse-grained ingots.

With few exceptions, nitrogen is determined by some variation of the Kjeldahl method, usually a micro or semi-micro procedure. In one instance, a spectrophotometric method using Nessler's reagent, is followed; in another, nitrogen gas volume is measured.

For metallics, quantitative spectrographic methods are most common. In some instances wet methods, or X-ray diffraction methods are used in conjunction with spectrographic methods, the choice of method used being influenced by the kinds and concentrations of elements sought.

In all of the foregoing analytical schemes, the unavailability of suitable standards has been recognized as a serious problem. At one facility, for example, uranium containing 410 ppm of carbon is the standard for carbon in tungsten. In others, NBS (National Bureau of Standards) carbon in steel standards are used. Silver oxide standards are used to calibrate apparatus for determining oxygen in tungsten. For nitrogen in tungsten, one facility uses an NBS nitrogen in titanium standard. In spectrographic work, synthetic or NBS standards are used. In many instances, the absence of satisfactory standards make the complete acceptance of analytical results difficult.

In discussions with various laboratory groups, it was evident that appreciable uncertainty exists in the reliability of all present analytical techniques for accurately measuring the impurities in tungsten in amounts below about 10 ppm. Several organizations[14,41] have shown that significant property changes can occur as a result of variation in impurity contents below the presently detectable limits. It is quite clear, therefore, that more work in improving the sensitivity and reliability of analytical techniques must be done in order to quantitatively assess the effects of various impurity elements on the properties of tungsten.

Mechanical Testing

Mechanical testing equipment in use is largely of the conventional type with some modifications for elevated-temperature testing. Both L. H. Marshall and Brew furnaces are in common use for this purpose. Temperature ceilings are, for the most part, in the 2000 to 3000 F range. One facility possessed vacuum creep-rupture and tensile equipment with a 4000 F ceiling. Another was using an electron-beam-heated stress-rupture unit operable "at temperatures above 5000 F, if necessary". In this last instance, the problem of exact temperature measurement has not been solved.

ACKNOWLEDGMENTS

In conducting this survey, all organizations were contacted which were known or believed to have had experience in preparing tungsten or tungsten-alloy compacts or ingots, in converting these to wrought forms, and/or in evaluating these materials for high-temperature mechanical properties. The cooperation of these organizations in contributing to this report is most sincerely appreciated.

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DJM/VDB/HRO:all

APPENDIX A

TUNGSTEN SURVEY QUESTIONNAIRE

AMC TUNGSTEN SHEET ROLLING PROGRAM
[CONTRACT AF 33(600)-41719]
STATE-OF-THE-ART SURVEY

I. Your Organization.

A. Are you a supplier of raw materials for use in producing tungsten?

Yes____ No____

A producer of mill shapes? Yes____ No____

A consumer? Yes____ No____

Other interest: Research____; Alloy development____; Other____

B. Have you made tungsten products by:

Arc melting? Yes____ No____

Electron beam melting? Yes____ No____

Powder metallurgy techniques? Yes____ No____

Other procedures? Yes____ No____. If "yes", please state procedure used: ____________________

II. Raw Materials.

A. What maximum levels of impurities of major importance are specified when procuring raw materials for use in the:

Consolidation of Tungsten by a Melting Procedure		Consolidation of Tungsten by a Powder Metallurgy Procedure	
________	________	________	________

B. What alloying and/or "doping" additions, if any, have been investigated?

1. Melting Procedure

Element or Compound	Quantity Added (or range of addition)	How Added
________	________	________

[Contract AF 33(600)-41719]

B-2. Powder Metallurgy Procedure

Element or Compound	Quantity Added (or range of addition)	How Added

C. What are the effects of alloying and "doping" additions noted in B (preceding page) on grain size, fabricability, and/or properties?

Element	Quantity Added (or range of additions)	Effect

D. Are you a supplier of tungsten raw materials? Yes____ No____

If yes:

Form	Size Range (for powders)

III. Consolidation.

A. Do you compact powder into shapes? Yes____ No____

If answer is "yes", what methods are used?

Compacting Method	Resulting Shape	Maximum Dimensions of Compacted Shape

B. What powder particle sizes and shapes are required for optimum compacting conditions?

Compacting Method	Average Particle Size	Particle Size Range	Particle Shape

C. What pressing procedures are used?

Pressure	Cold-Pressed Density	Lubricant Used (if any)	Die Shape

Additional comments on uniformity of density in cold-pressed shapes:

____

[Contract AF 33(600)-41719]

III. -D. What sintering procedures are used?

	For Electrodes	For Powder Metallurgy Process
Temperature	________	________
Time	________	________
Atmosphere	________	________
Resulting as-sintered density	________	________

E. What critical chemical reactions, if any, occur on sintering?

F. Where electrodes are produced for subsequent arc melting, how are sections joined?

G. What electrode-to-mold size ratios are used in arc melting?

H. What electrode configurations are used?

I. What are the optimum melting conditions according to your experience?

Electrode/mold ratio ________

Ingot size ________

Voltage ________

AC or DC ________

Amperage ________

Furnace atmosphere ________

Pressure above melt ________

J. Do you use any unusual processes in melting, differing from those commonly used in arc-melting practice? (mold liners, arc initiation, etc.)

[Contract AF 33(600)-41719]

III. -K. What is the maximum size of unalloyed tungsten arc-cast ingot which you can supply?

L. What is your normal yield on conditioned arc-melted ingots?

M. What are your electron-beam melting capabilities?

N. What methods are used for inspection of your consolidated product?

Inspection Method	Consolidated From Powder	Consolidated by Arc Melting
______	______	______

O. Please discuss any other means of consolidation not listed above, which you have employed:

IV. Fabrication.

A. Powder Metallurgy.

1. What are the maximum size wrought shapes you have made?

Forgings ______________________

Extrusions ______________________

Rolled bar ______________________

Sheet: gage	width	length
<0.020"	____	____
0.020"	____	____
0.040"	____	____
0.060"	____	____
>0.060"	____	____

[Contract AF 33(600)-41719]

IV. -A-2. What conditions are used in initial breakdown for the production of sheet?

Method of Mechanical Working	Size of Workpiece	Preheating Temperature	Preheating Atmosphere	Lubricant (if any)
______	______	______	______	______

Extrusion ratio (if extrusion is used) ____________________

____

3. What reduction schedules are used in forging and rolling to sheet?

Working Operation	Preheating Temperature	Amount of Reduction Per Pass	Amount of Reduction Between Anneals	Annealing Temperature	Annealing Atmosphere
______	______	______	______	______	______

4. What is the size and separating force of your rolling mill?

____

5. Do you use a protective atmosphere or protective coating?

Yes____ No____. Composition of atmosphere or coating,

if used: ____

6. What surface conditioning treatments are used?

	Pickling	Grinding	Other
Intermediate	______	______	______
Final	______	______	______

7. What straightening or flattening procedures are used for final sheet?

[Contract AF 33(600)-417`9]

IV. -B. Arc-Cast (or Otherwise Melted) Material.

1. What are the maximum size wrought shapes you have made?

Forgings ____________________

Extrusions ____________________

Rolled bar ____________________

Sheet: gage	width	length
<0.020"	______	______
0.020"	______	______
0.040"	______	______
0.060"	______	______
>0.060"	______	______

2. What conditions are used in initial breakdown for the production of sheet?

Method of Mechanical Working	Size of Workpiece	Preheating Temperature	Preheating Atmosphere	Lubricant (if any)
______	______	______	______	______

Extrusion ratio (if extrusion is used) ____________________

____

3. What reduction schedules are used in forging and rolling to sheet?

Working Operation	Preheating Temperature	Amount of Reduction Per Pass	Amount of Reduction Between Anneals	Annealing Temperature	Annealing Atmosphere
______	______	______	______	______	______

4. What is the size and separating force of your rolling mill?

____

____

[Contract AF 33(600)-41719]

IV. -B-5. Do you use a protective atmosphere or protective coating?

Yes____ No____ Composition of atmosphere or coating, if

used: ____

____

6. What surface conditioning treatments are used?

	Pickling	Grinding	Other
Intermediate	______	______	____
Final	______	______	____

7. What straightening or flattening procedures are used for final sheet?

C. What are the significant variables in fabricating arc-cast vs. powder compacted shapes?

V. (a) What types of mechanical testing equipment do you have, and what are their limitations?

(b) What are your chemical analysis procedures and standards?

For carbon ____

____

For oxygen ____

____

For nitrogen ____

____

For metallics ____

____

DATA SHEET FOR TUNGSTEN AND TUNGSTEN-BASE ALLOYS

Please include developmental alloys. For proprietary alloys please consider listing data without specifying alloy content.

Company ______________________________

Alloy Designation ______________________________

Composition ______________________________

This alloy is ____ experimental ______ pilot plant ________ commercial.

Method of Consolidation ______________________________

Initial Breakdown by ______________________________

Subsequent Fabrication by ______________________________

What special procedures or precautions are necessary in fabrication? ______________

____

Properties:

Recrystallization Temperature (specify amount of work and working temperature in piece tested.)

____

Tensile Properties

Tensile Data at Strain Rate of ______in/in/min. in ______ atmosphere.

Condition	Test Temperature	Yield Strength (Kips)	Ultimate Tensile Strength (Kips)

% Elongation	% Reduction of Area	Elastic Modulus	Hardness

DATA SHEET FOR TUNGSTEN AND TUNGSTEN-BASE ALLOYS

Creep and/or Stress Rupture Data:

Transition Temperature (specify type and testing conditions):

Oxidation Data:

Additional Comments:

Please attach reprints, if available, or references to published or inhouse reports or data sheets where these or additional property data on tungsten and tungsten alloys are available.

APPENDIX B

ORGANIZATIONS CONTACTED IN SURVEY

APPENDIX B

ORGANIZATIONS CONTACTED IN SURVEY

Aerojet General Corporation
Azusa, California

Aerojet General Corporation
Sacramento, California

Aerospace Industries Association
Los Angeles 36, California

*Air Materiel Command
Ohio

Allegheny Ludlum Steel Corporation
Brackenridge, Pennsylvania

Allegheny Ludlum Steel Corporation
Watervliet, New York

Alloyd Corporation
Cambridge 39, Massachusetts

Arcturus Manufacturing Corporation
Venice, California

Armour Research Foundation
Chicago 16, Illinois

Babcock and Wilcox Company
Beaver Falls, Pennsylvania

Bell Aerosystems Company
Buffalo 5, New York

Bell Helicopter Corporation
Fort Worth 1, Texas

Bendix Aviation Corporation
South Bend 20, Indiana

Boeing Airplane Company
Seattle 24, Washington

Boeing Airplane Company
Wichita 1, Kansas

*Bureau of Mines
Albany, Oregon

Bureau of Mines
Rolla, Missouri

Bureau of Naval Weapons
Washington 25, D. C.

Bureau of Ships
Washington 25, D. C.

California Institute of Technology
Pasadena, California

Cameron Iron Works, Inc.
Houston 1, Texas

Canton Drop Forging and Manufacturing Company
Canton, Ohio

Chance Vought Aircraft, Inc.
Dallas, Texas

Chromalloy Corporation
White Plains, New York

Cleveland Tungsten, Inc.
Cleveland 5, Ohio

Clevite Research Center
Cleveland, Ohio

*Climax Molybdenum Company of Michigan
Detroit 3, Michigan

Convair
Fort Worth, Texas

Convair
Pomona, California

Convair
San Diego 12, California

*Designates organization visited.

Convair Astronautics Division
San Diego 12, California

Crucible Steel Company of America
Midland, Pennsylvania

Curtiss-Wright Corporation
Buffalo 15, New York

Curtiss-Wright Corporation
Wood Ridge, New Jersey

Denver Research Institute
Denver 10, Colorado

Douglas Aircraft Company, Inc.
El Segundo, California

Douglas Aircraft Company, Inc.
Long Beach, California

Douglas Aircraft Company, Inc.
Santa Monica, California

Douglas Aircraft Company, Inc.
Tulsa 15, Oklahoma

E. I. du Pont de Nemours & Company
Wilmington 98, Delaware

Eastern Stainless Steel Corporation
Baltimore, Maryland

Fairchild Engine and Airplane Corporation
Oak Ridge, Tennessee

*Fansteel Metallurgical Company
North Chicago, Illinois

*Firth Sterling Incorporated
Pittsburgh 30, Pennsylvania

Frankford Arsenal
Philadelphia, Pennsylvania

General Electric Company
Cincinnati 15, Ohio

General Electric Company
Cincinnati 15, Ohio

General Electric Company
Cincinnati 15, Ohio

*General Electric Company
Cleveland 17, Ohio

General Electric Company
Evendale, Ohio

General Electric Company
Schenectady, New York

General Motors Corporation
Indianapolis, Indiana

General Motors Corporation
Warren, Michigan

General Telephone & Electronic Labs, Inc.
Bayside, New York

Grumann Aircraft Corporation
Bethpage, L. I., New York

Harvey Aluminum, Incorporated
Torrance, California

Haynes Stellite Company
Kokomo, Indiana

Hughes Tool Company
Culver City, California

Kelsey-Hayes Company
New Hartford, New York

Kennametal, Incorporated
Latrobe, Pennsylvania

Kennecott Copper Corporation
New York 17, New York

Kropp Forge Company
Chicago 50, Illinois

Kulite Tungsten Company
Ridgefield, New Jersey

Ladish Company
Cudahy, Wisconsin

* Designates organization visited.

Linde Company
Indianapolis 24, Indiana

Lockheed Aircraft Corporation
Burbank, California

Lockheed Aircraft Corporation
Marietta, Georgia

Lockheed Aircraft Corporation
Palo Alto, California
Metallurgy and Ceramics Research

Lockheed Aircraft Corporation
Palo Alto, California

Lockheed Aircraft Corporation
Sunnyvale, California

Lockheed Aircraft Corporation
Van Nuys, California

Los Alamos Scientific Laboratory
Los Alamos, New Mexico

Lycoming Division
Stratford, Connecticut

Marquardt Aircraft Company
Ogden, Utah

Marquardt Aircraft Company
Van Nuys, California

Martin Company
Baltimore 3, Maryland

Martin Company
Denver 1, Colorado

Martin Company
Orlando, Florida

Massachusetts Institute of Technology
Cambridge 39, Massachusetts

Materials Advisory Board
Washington 25, D. C.

McDonnell Aircraft Corporation
St. Louis 3, Missouri

Metallwork Plansee AG
Reutte, Tyrol

*National Aeronautics and Space Admin.
Cleveland 35, Ohio

National Aeronautics and Space Admin.
Washington 25, D. C.

National Bureau of Standards
Washington 25, D. C.

National Research Corporation
Cambridge 42, Massachusetts

Naval Air Material Center
Philadelphia 12, Pennsylvania

Naval Research Laboratory
Washington 25, D. C.

New York University
New York 53, New York

North American Aviation, Inc.
Canoga Park, California

North American Aviation, Inc.
Columbus, Ohio

North American Aviation, Inc.
Downey, California

North American Aviation, Inc.
Los Angeles 45, California

North American Philips Company, Inc.
Lewiston, Maine

Northrop Aircraft Corporation
Hawthorne, California

Office of Naval Research
Washington 25, D. C.

Office of Ordnance Research
Durham, North Carolina

*Oregon Metallurgical Corporation
Albany, Oregon

*Designates organization visted.

Philips Metalonics
New York

Pratt & Whitney Aircraft
Middletown, Connecticut

Raytheon Company
Andover, Massachusetts

Reactive Metals, Inc.
Niles, Ohio

Reduction and Refining Company
Kenilworth, New Jersey

Reisner Forge Company
Southgate, California

Rembar Company, Incorporated
Dobbs Ferry, New York

Republic Aviation Corporation
Farmingdale, L. I., New York

Rohr Aircraft Corporation
Chula Vista, California

Ryan Aeronautical Company
San Diego 12, California

Sandia Corporation
Albuquerque, New Mexico

Sandia Corporation
Livermore, California

Shieldalloy Corporation
Newfield, New Jersey

Solar Aircraft Company
San Diego 12, California

Southern Research Institute
Birmingham, Alabama

Stanford Research Institute
Palo Alto, California

Stauffer Metals Company
Richmond, California

*Steel Improvement and Forge Company
Cleveland, Ohio

Super-Temp Engineering and
Manufacturing, Inc.
Long Beach 13, California

Sylvania Electric Products, Inc.
Towanda, Pennsylvania

Taylor Forge and Pipe Works
Chicago 90, Illinois

Temco Aircraft Corporation
Dallas 22, Texas

Temescal Metallurgical Corporation
Richmond, California

Thermionic Products Company
Plainfield, New Jersey

Thiokol Chemical Corporation
Denville, New Jersey

*Thompson Ramo Wooldridge, Inc.
Cleveland 17, Ohio

*Union Carbide Metals Company
Niagara Falls, New York

Union Carbide Nuclear Company
Oak Ridge, Tennessee

Union Carbide Nuclear Corporation
Oak Ridge, Tennessee

United Aircraft Corporation
East Hartford 8, Connecticut

*Universal-Cyclops Steel Corporation
Bridgeville, Pennsylvania

University of California, Radiation
Laboratory
Livermore, California

*Wah Chang Corporation
Albany, Oregon

*Designates organization visited.

Wah Chang Corporation
New York 7, New York

Watertown Arsenal Laboratory
Watertown, Massachusetts

*Westinghouse Electric Corporation
Blairsville, Pennsylvania

Westinghouse Electric Corporation
Bloomfield, New Jersey

Westinghouse Electric Corporation
Kansas City, Missouri

Westinghouse Laboratories
Pittsburgh 35, Pennsylvania

*Wright Air Development Division
Dayton, Ohio

Wyman-Gordon Company
North Grafton, Massachusetts

*Designates organization visited.